Naval Facilities Engineering Command Southwest
BRAC PMO West
San Diego, California

DRAFT

PARCEL E REMOVAL SITE EVALUATION SAMPLING AND ANALYSIS PLAN

RADIOLOGICAL CONFIRMATION SAMPLING AND SURVEY AT PARCEL E

FORMER HUNTERS POINT NAVAL SHIPYARD SAN FRANCISCO, CALIFORNIA

October 2021

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DCN:

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October 2021

Prepared for:

Department of the Navy
Naval Facilities Engineering Command Southwest
1220 Pacific Highway
San Diego, CA 92132

Prepared by: EIP, Burlingame, CA
Contract Number: N62473-17-D-0011; Task Order No. N6247319F5198
DCN:

SAP WS #1: TITLE AND APPROVAL PAGE

DRAFT

SAMPLING AND ANALYSIS PLAN –
Field Sampling Plan and Quality Assurance Project Plan

October 2021

Parcel E - Removal Site Evaluation
Former Hunters Point Naval Shipyard
San Francisco, California

Prepared for:

Base Realignment and Closure
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Review Signature: _____
Rich Gioscia, Program Quality Control Manager Date

Approval Signature: _____
Judy Solomon NAVFAC, Quality Assurance Officer Date

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EXECUTIVE SUMMARY

This document is Sampling and Analysis Plan (SAP) based on the *Final Parcel G Removal Site Evaluation, Sampling and Analysis Plan, Former Hunters Point Naval Shipyard, San Francisco*. ECC Insight Philotechnics, LLC (EIP) has prepared this Parcel E Sampling and Analysis Plan (SAP) in support of the confirmation site investigation to determine whether current site conditions are compliant with the remedial action objective in the *Record of Decision for Parcel E, Hunters Point Shipyard, San Francisco, California* (U.S. Department of the Navy [Navy], 2013).

Sites addressed under this SAP include former radiologically impacted areas in Parcel E, which occupies approximately 128 acres in the southwest portion of the Former Hunters Point Naval Shipyard. Radiological surveys and remediation were previously conducted at Hunters Point Naval Shipyard as part of a base wide time-critical removal action. Tetra Tech EC, Inc., (TtEC) under contracts with the Navy, conducted a large portion of the base wide time-critical removal action, including Parcel E. Data manipulation and falsification were committed by TtEC employees during the time-critical removal action. An independent third-party evaluation of previous data identified additional potential manipulation and falsification at Parcel E and data quality issues with data collected (Navy, 2017 and 2018). As a result, the Navy will conduct investigations at radiologically impacted soil and building sites in Parcel E that were surveyed by TtEC. Soil investigations will be conducted in a phased approach at the following areas in Parcel E. Soil investigation areas will be divided into trench units and surface soil survey units. The size and boundary of the trench units and survey units will be based on previous plans and reports.

- Trench Units: Former Sanitary Sewer and Storm Drain Trenches
- Former Building Site Surveys and Survey Units: 517, 503, 520, 506, 508, 510/510A, 509, 529, IR-04 Former Scrap Yard/Former building 807 Site, 704, 704 Storage Yard, and Former Shacks 79 and 80
- Existing Building Surveys: 406, 414, 521, 810

This Parcel E SAP will be used as a reference document by field and laboratory(s) personnel engaged in sampling and analysis for the Parcel E project. This document will be provided to individuals listed in SAP Worksheets (WSs) #3 and #4.

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Attachment 2 Laboratory Standard Operating Procedures and Certification (*provided on electronic copy only*)
Attachment 3 EIP Standard Operating Procedures *(provided on electronic copy only)*

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List of Acronyms

%	percent
<	less than
>	greater than
±	plus or minus
≤	less than or equal to
≥	greater than or equal to
σ	sigma
°C	degree Celsius
^{60}Co	cobalt-60
^{90}Sr	strontium-90
^{137}Cs	cesium-137
^{226}Ra	radium-226
^{234}U	uranium-234
^{235}U	uranium-235
^{238}U	uranium-238
^{238}Pu	plutonium-238
^{239}Pu	plutonium-239
^{240}Pu	plutonium-240
ANSI	American National Standards Institute
BRAC	Base Realignment and Closure
BSC	background subtraction count
CARB	California Air Resources Board
CAS	Chemical Abstracts Service
CCV	continuing calibration verification
CDPH	California Department of Public Health
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980
CM	Construction Manager
COC	Chain-of-Custody
COR	Contracting Officers Representative
DER	duplicate error ratio
DL	detection limit (same as Method Detection Limit)
DLC	decision level concentration
DoD	U.S. Department of Defense
DQA	data quality assessment
DQI	data quality indicator
DQO	data quality objective
DTSC	California Department of Toxic Substances Control
DRO	Diesel Range Organics
ECC	Environmental Chemical Corporation
EIP, LLC	ECC Insight Philotechnics, LLC
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
ESU	excavated soil unit
FWHM	full width at half maximum
GFPC	gas flow proportional counting
GRO	Gasoline Range Organics
H&S	health and safety

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HPNS	Hunters Point Naval Shipyard
HRA	Historical Radiological Assessment
ICAL	initial calibration
ICALE	initial calibration - efficiency
ICV	initial calibration - verification
IDW	Investigative Derived Waste
IL	Investigation Level
keV	kiloelectron volt
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	Limit of Detection
LOQ	Limit of Quantitation
LRSO	Licensed Radiological Safety Officer
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols Manual
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MB	method blank
MDC	minimum detectable concentration
MDL	Method Detection Limit (same as detection limit)
mg/kg	milligrams per kilogram
MRO	Motor Oil Range Organics
MS	matrix spike
MSD	matrix spike duplicate
MQO	Method Quality Objectives
NAVFAC SW	Naval Facilities Engineering Command Southwest
NAVSEA	Naval Sea Systems Command
NAVSEADET	Naval Sea System Detachment
Navy	U.S. Department of the Navy
NORM	naturally occurring radioactive material
NRDL	Navy Radiological Defense Laboratory
OSHA	Occupational Safety and Health Administration
PARCCS	precision, accuracy, representativeness, completeness, comparability, and sensitivity
pCi/g	picocuries per gram
PM	project manager
PgQCM	Program Quality Control Manager
POC	point of contact
PQCM	Project Quality Control Manager
QA	quality assurance
QAO	Quality Assurance Officer
QC	quality control
QCO	quality control officer
QL	quantitation limit
QSM	Quality Systems Manual
RACR	remedial action completion reports
RAO	remedial action objective
RASO	Radiological Affairs Support Office
RCT	Radiological Control Technician
RG	remediation goal
ROC	radionuclide of concern
ROD	record of decision

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ROICC	Regional Officer in Charge of Construction
ROM	Radiological Operations Manager
RPD	relative percent difference
RPM	remedial project manager
RSO	radiation safety officer
RSY	radiological screening yard
S3	soil sorting system
SAP	sampling and analysis plan
SD	storm drain
SFDPH	San Francisco Department of Public Health
SFU	sidewall floor unit
SOP	standard operating procedure
SS	sanitary sewer
SSHO	Site Safety and Health Officer
SU	survey unit
TPH	Total Petroleum Hydrocarbons
TtEC	Tetra Tech EC, Inc.
TU	trench unit
VSP	Visual Sample Plan
Water Board	California Regional Water Quality Control Board
WP	work plan
WS	worksheet

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SAP WS #2: SAP IDENTIFYING INFORMATION

Site Name/Number: **Hunters Point Naval Shipyard (HPNS)**
Parcel: **E**
Contractor Name: **EIP, LLC**
Contract Number: **N62473-15-D-0811, X031**
Contract Title: **RADIOLOGICAL CONFIRMATION SAMPLING AND SURVEY AT PARCEL E, FORMER HUNTERS POINT NAVAL SHIPYARD, SAN FRANCISCO, CALIFORNIA**

Work Assignment Number (optional): -

This sampling and analysis plan (SAP) was prepared in accordance with Naval Facilities Engineering Command Southwest (NAVFAC SW) work instructions and the following guidance documents:

- *Guidance for Quality Assurance Project Plans* (U.S. Environmental Protection Agency [EPA], 2002)
- *Uniform Federal Policy for Quality Assurance Project Plans, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs* (EPA, 2005)
- *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006)
- *Quality Systems Manual for Environmental Laboratories, Version 5.3* (QSM; U.S. Department of Defense [DoD], 2019)

Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).

This is a project -specific SAP.

Kick-off Meeting on Nov 17 and 22, 2016 (See worksheet #9)

Additional Bi-weekly meetings

- The U.S. Department of the Navy (Navy) Base Realignment and Closure (BRAC) Project Management Office held project kickoff meetings on November 17 and 22, 2016, and a meeting with the regulators, including EPA, California Department of Toxic Substances Control (DTSC), Office of Community Investment and Infrastructure, San Francisco Department of Public Health (SFDPH), and California Regional Water Quality Control Board, San Francisco Bay Region (Water Board) on December 7, 2016.

- The Navy assembled a Technical Team (a group of technical experts) that includes representatives from the Navy, EPA, DTSC, California Department of Public Health (CDPH), and the City of San Francisco. The Technical Team conducted an evaluation of previous HPNS data in light of the claims made and is developing an approach for follow-up investigations. The Technical Team meets at least bi-weekly to discuss project updates and review documents. To date, several work plan (WP) iterations have been submitted and reviewed. For soil, a phased approach was designed based on a proposal by the regulatory agencies on an initial draft WP. For buildings, the approach was designed based on regulatory comments on an initial draft WP to conduct surveys based on the Parcel G Record of Decision (ROD) (Navy, 2009a, 2009b). The approaches for soil and buildings are included in the *Final Parcel G Removal Site Evaluation, Sampling and Analysis Plan, Former Hunters Point Naval Shipyard, San Francisco, CA*, (CH2M Hill, 2019).

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Title	Date
None	

List organizational partners (stakeholders) and connection with lead organization:

DTSC– Regulatory Stakeholder
CDPH– Regulatory Stakeholder
Water Board– Regulatory Stakeholder
EPA– Regulatory Stakeholder
City of San Francisco—Future Property Owner

Lead organization

U.S. Department of the Navy (Navy) – NAVFAC Southwest, BRAC Program Management Office West

If required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

No worksheets (WSs) were omitted from this SAP

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SAP WS #3: DISTRIBUTION LIST

Name of SAP Recipients	Title/Role	Organization	Telephone Number	Email Address
Paul Stoick	Navy COR	NAVFAC SW	619-524-5755	paul.stoick@navy.mil
Sean-Ryan McCray	Navy Lead Remedial Project Manager (RPM)	NAVFAC SW	619-507-2949	Sean-ryan.mccray.ctr@navy.mil
Matt Liscio	Navy Radiological Affair and Safety Officer	NAVSEADET RASO	757-887-4692	navsearasoadmin.fct@navy.mil
Shirley Ng / Hamid Naimi	Regional Officer in Charge of Construction (ROICC) Project Engineer	NAVFAC SW	510-501-1170	shirley.ng@navy.mil hamid.naimi@navy.mil
Judy Solomon	Quality Assurance Officer (QAO)	NAVFAC – Atlantic	757-322-4744	Judith.A.Solomon7.civ@us.navy.mil
Doug Delong	Certified Safety Officer (CSO)	NAVFAC SW	415-743-4713 (office) 510-220-1894 (mobile)	douglas.delong.ctr@navy.mil
Karen Ueno	USEPA RPM	United States Environmental Protection Agency	415-972-2480	ueno.karen@epa.gov
Nina Bacey	DTSC RPM	California Department of Toxic Substances	510-540-2480	juanita.bacey@dtsc.ca.gov
Sheetal Singh	CDPH Project Manager (PM)	California Department of Public Health	916-449-5691	sheetal.singh@cdph.ca.gov
Amy Brownell	SFDPH RPM	San Francisco Department of Public Health	415-252-3967	amy.brownell@sfdph.org
Tina Low	RWQCB RPM	California Regional Water Quality Control Board	510-622-5682	tina.low@waterboards.ca.gov

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Name of SAP Recipients	Title/Role	Organization	Telephone Number	Email Address
Matt Long	Project Manager (PM)	EIP	774-244-7102 (cell) 303-298-7607 (office)	mlong@ecc.net
Joe Edie	Construction Manager (CM)	EIP	650-464-6033 (cell) 650-347-1555 (office)	jerdie@ecc.net
Rich Gioscia	Program Quality Control Manager (PgQCM)	EIP	303-898-8859 (cell) 303-298-7607 (office)	rgioscia@ecc.net
Rinku Shah	Project Quality Control Manager (PQCM)	EIP	-	rshah@ecc.com
Keith Anderson	Radiological Operations Manager (ROM)	EIP	303-356-1267 (cell)	Kanderson@ecc.net
Alejandro (Alex) Lopez	Licensed Radiation Safety Officer (LRSO)	Perma-Fix	970-778-0449	alopez@perma-fix.com
Theodore Johnson	Site Safety Health Officer (SSHO) / Radiation Safety Officer (RSO)	EIP	303-472-8834 (cell)	tjohnson@ecc.net
Jackson Kiker	Program Chemist	EIP	508-229-2270 (office) 774-245-0904 (cell)	jkiker@ecc.net
Kane McNeill	Project Chemist (PC)	EIP	650-228-6950	kmcneill@ecc.net
TBD	Field Geologist	EIP	-	-
Rhonda Ridenhower	Laboratory PM	Eurofins Test America – St. Louis, MO	314-787-8227	Rhonda.Ridenhower@testamericainc.com
Nick Ly	Laboratory PM	NVL Laboratory	206-547-0100	nick.l@nvllabs.com
Christina Rink-Ashdown	Data Validation PM	Laboratory Data Consultants, Inc	760-827-1100	crink@lab-data.com

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SAP WS #4: PROJECT PERSONNEL SIGN-OFF SHEET

Name	Organization/Title/Role	Signature/Email Receipt	Date SAP Read
Matt Long	EIP / Project Manager		
Joe Erdie	EIP / Construction Manager		
Rinku Shah	EIP / Project QC Manager		
Keith Anderson	EIP / Radiological Operations Manager		
Alejandro Lopez	Perma-Fix / Licensed Radiation Safety Officer		
Ted Johnson	EIP / SSHO & RSO		
Jackson Kiker	EIP / Program Chemist		
Kane McNeill	EIP / Project Chemist		
Rhonda Ridenhour	Eurofins Test America / PM / Lab Analyses		
Christina Rink-Ashdown	Laboratory Data Consultants / PM / Data Validation		

Note:
The signed WS#4 will be stored either on-site in the project files or as a PDF on the ECC's project electronic Sharedrive

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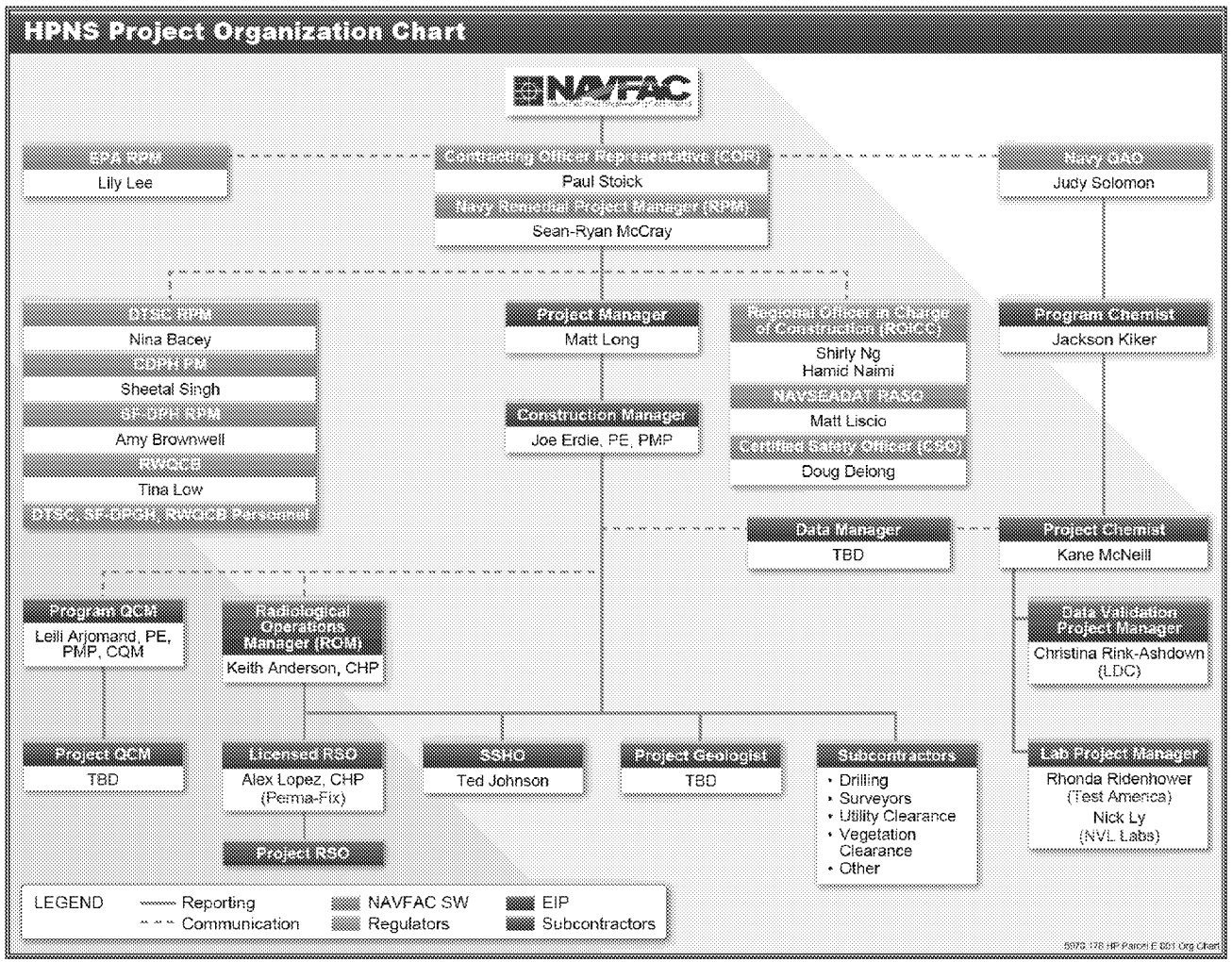
SAP WS #5: PROJECT ORGANIZATION CHART



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SAP WS #6: COMMUNICATION PATHWAYS

Communication Drivers	Organization	Title/Role	Name	Telephone Number	Procedure
Communication with Regulators	Navy	Navy RPM	Sean-Ryan McCray	619-507-2949	The Navy RPM will be the primary point of contact (POC) for communication with regulators for project information. The EIP, LLC Senior PM will communicate project field changes, construction, or quality issues to the Navy RPM. The Navy will notify EPA, DTSC, WB, CDPH, and SFDPH by email or telephone for significant field changes effecting the scope or implementation of the design. The Navy RPM will communicate project field changes and construction or quality issues, by email or telephone, to the regulators within five days.
POC with Navy Lead RPM or Lead Assistant RPM	EIP	Project Manager Construction Manager	Matt Long Joe Erdie	774-244-7102 (cell) 650-464-6033 (cell)	Relevant materials and information about the project will be forwarded to Navy Lead RPM and Lead Assistant RPM by email or telephone by the Senior PM or CM within three business days.
POC with the Navy RSO	EIP	Construction Manager Radiological Operations Manager	Joe Erdie Keith Anderson	650-464-6033 (cell) 303-356-1267 (cell)	The CM or RSO will be the primary POC for communication with the Navy RASO regarding radiological issues.
SAP Review and Approval	EIP	Program Chemist	Jackson Kiker	508-229-2270 (office) 774-245-0904 (cell)	The Program Chemist will be the primary POC for communication with the Navy QAO regarding SAP review and approvals according to standard document review and approval timelines. Works with Navy QAO to assure the SAP provided meets the requirements necessary so the review and approval times may meet the Standard Navy SAP timeline.

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Communication Drivers	Organization	Title/Role	Name	Telephone Number	Procedure
SAP Amendments or Changes	EIP	Program Chemist Project Chemist	Jackson Kiker Kane McNeill	508-229-2270 (office) 774-245-0904 (cell)- 650-228-6950	The Project Chemist is responsible for documenting field changes related to sampling and for informing or seeking approval from the Program Chemist or Navy QAO. The Program or Project Chemist is also responsible for generating SAP amendments as necessary for approval by the Navy QAO. The Program or Project Chemist oversees the documentation, notification, and corrective actions associated with data collection issues. Due to the potential impact field changes and SAP amendments may have on the project, the Project Chemist is to be notified by email or telephone of such issues within 24 hours.
Stop Work Issues (Quality)	Navy	Navy QAO	Judy Solomons	757-322-4744	The Navy QAO is authorized to suspend project execution if quality assurance (QA) requirements are not adequately followed. The Navy QAO will notify the Navy RPM and ECC Program QC Manager by email or telephone if QC issues requiring stop work are identified. The ECC Program QC Manager or Program Chemist, in consultation with the Task Order Manager and Navy RPM, will work with the Navy QAO to resolve QA issues and resume work. Upon resolution, the Project QC Manager oversees the documentation, notification, and corrective actions associated with site issues in writing within five business days.
Health and Safety (H&S) Issues	EIP	Site Safety and Health Officer (SSHO)	Theodore Johnson	303-472-8834 (cell)	The Project SSHO is the POC for H&S issues. If H&S issues are not resolved at the project level (in consultation with the PM, Site Construction Manager, Technical Lead, etc.), then the issue will be elevated to the Program SSHO. The Program SSHO or designee will seek additional guidance or approval from the Navy H&S Officer, if necessary. Upon resolution, the Project SSHO oversees the documentation, notification, and corrective actions associated with the issue in writing. Due to the potential seriousness of H&S issues, the SSHO is to be notified in person or by telephone of H&S issues immediately.

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Communication Drivers	Organization	Title/Role	Name	Telephone Number	Procedure
Sample Collection Issues	EIP	Primary: Project Chemist Secondary: Program Chemist	Kane McNeill Jackson Kiker	650-228-6950 508-229-2270 (office) 774-245-0904 (cell)	The Project Chemist (PC) is the POC for sampling and chemistry issues. If sampling issues are not resolved at the project level (in consultation with the PM, Construction Manager, Technical Lead, Geologist, etc.), then the issue will be elevated to the Program Chemist or Program QC Manager. The Program Chemist or Program QC Manager will seek additional guidance or approval from the Navy QAO, if necessary. Upon resolution, the PC oversees the documentation, notification, and corrective actions associated with the QA issue in writing. Due to the potential impact, sampling issues may have on the project the PC is to be notified of sampling issues by email or telephone within 24 hours.
Laboratory Reporting or Data Quality Issues	EIP	Project Chemist	Kane McNeill	650-228-6950	The Project Chemist (PC) is the POC for laboratory issues and electronic data deliverables (EDDs). If laboratory issues are not resolved with the PC or data manager, then the issue will be elevated to the Program Chemist. If significant problems are identified by the project chemist, data validation project manager, or laboratory project managers that will impact the usability of the data, then the Project Chemist should inform the PM, Navy RPM, Navy QAO, Program Chemist by email or telephone within 24 hours of discovery or by the next business day. Upon resolution, the PC oversees the documentation, notification, and corrective actions associated with the laboratory issue in writing. Laboratory reporting issues will be corrected within five business days.
Construction or Sampling Quality Issues	EIP	Project QC Manager Program Chemist Program QC Manager	Rinku Shah Jackson Kiker Leili Arjomand	973-738-2513 508-229-2270 (office) 774-245-0904 (cell) 650-347-1555 (office) 650-208-5482 (cell)	In general, the PC is the POC for sampling and chemistry issues and the Project QC Manager is the POC for other quality issues. If quality issues are not resolved at the project level (in consultation with the PM, site supervisor, technical lead, etc.), then the issue will be elevated to the Program QC Manager or the Program Chemist. The Program QC Manager or Program Chemist will seek additional guidance or approval from the Navy QAO, if necessary. Upon resolution, the Project QC Manager or PC oversees the documentation, notification, and corrective actions associated with the QA issue in writing within five days.

Communication Drivers	Organization	Title/Role	Name	Telephone Number	Procedure
Notification of Non-Usable Analytical Data	EIP	Program Chemist	Jackson Kiker	508-229-2270 (office) 774-245-0904 (cell)	If significant problems are identified by the laboratory or the project team that impact the usability of the data (i.e., the data is rejected or the data quality objectives [DQOs] are not met), then the Program Chemist will notify the NAVFAC SW RPM and the Navy QAO by email or telephone within 24 hours of notification from the project chemist or the next business day.
Immediate Stop Work Issues (H&S)	EIP	SSHO On-site Employees	Ted Johnson All Employees	303-472-8834 (cell) -	Employees have the right and duty to stop work immediately when conditions are unsafe, or when established safety procedures are being disregarded. Whenever an employee determines that workplace conditions present an immediate uncontrolled risk of injury or illness, immediate resolution with the appropriate supervisor shall be sought. Should the supervisor be unable or unwilling to correct the unsafe conditions, the employee or H&S Officer is authorized and required to issue a Stop Work Order. The specific activity or operation in question shall be discontinued until the issue is resolved. Stop work issues originating from personnel in the field, would first inform the on-site project personnel affected by the issue, followed by the SSHO/ROM/CM/PM, then the SSHO, ROM, CM, or PM would inform the Navy RPM.

SAP WS #7: PERSONNEL RESPONSIBILITIES AND QUALIFICATIONS TABLE

Name	Title/Role	Organizational Affiliation	Responsibility
Sean McCray	Navy RPM	Navy	Manages governmental oversight of the project Manages project funding and scope Coordinates project documents review Primary contact and liaison with regulatory agencies Responsible for technical oversight of the project
Judy Solomon	QAO	Navy	Provides governmental oversight of the EIP QA program Provides quality-related directives through Contracting Officer's Technical Representative Provides technical and administrative oversight of EIP surveillance audit activities Reviews and approves the SAP prior to regulatory review or field implementation Acts as POC on matters concerning QA and the client's laboratory QA program Authorized to suspend project execution if QA requirements are not adequately followed
Matt Long	Senior PM	EIP	Manages oversight of the project for EIP Primary POC for communication with the Navy RPM and Navy Contracts Ensures that requirements of project contract are attained in a manner consistent with project plans Oversees planning, execution, and conclusion of project activities Manages project budgets and schedules
Joe Erdie	Construction Manager	EIP	Secondary POC for communication with the Navy RPM and Navy Contracts Ensures that requirements of project contract are attained in a manner consistent with project plans Daily oversight of planning, execution, and conclusion of project activities
Leili Arjomand	Program QC Manager	EIP	Oversight/Review of: - Inspections performed by Project QC Manager - Project QC Criteria outlined in project plans Develops the project QC objectives and prepares the construction QC plan Serves as a technical advisor to the project
Rinku Shah	Project QC Manager	EIP	Conducts inspections (preparatory, initial, follow up, completion) Develops the project QC objectives and prepares the construction QC plan Daily Administration of the QC plan Provides training on QC objectives as required to on-site project personnel Manages QC documentation and QC deliverables Lists definable features of work Confirms data collected from field activities will support NIRIS requirements

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Name	Title/Role	Organizational Affiliation	Responsibility
Keith Anderson	Radiological Operation Manager	EIP	Oversight of LSRO Reviewing and approving radiological WPs and final reports. Providing review and concurrence on data for proposed radiological actions Comparing radiological data with the requirements of the WP, design plans, task specific plans, and SAP to ensure that proper conditions have been met to implement the action requested Ensuring that the radiological data reported are consistent with the intent for which the data were provided Reviewing uncertainty counting and the 2 sigma (σ) total uncertainty data along with the laboratory qualifiers to determine if the data are of sufficient quality
Alejandro Lopez	Licensed Radiological Safety Officer	Perma-Fix	Secondary: Reviewing radiological laboratory data on a routine basis Providing review and concurrence on data for proposed radiological actions Comparing radiological data with the requirements of the WP, design plans, task-specific plans, and SAP to ensure that proper conditions have been met to implement the action requested Ensuring that the radiological data reported are consistent with the intent for which the data were provided Reviewing uncertainty counting and the 2 sigma (σ) total uncertainty data along with the laboratory qualifiers to determine if the data are of sufficient quality
Ted Johnson / TBD	Radiological Safety Officer	EIP	Oversees daily overall radiological operations and documentation for the project Acts as the technical lead for radiological data collection Ensures that the Project Radiological Control Technicians have adequate training in sample collection Secondary: Receives and reviews QA laboratory sample data (Data Validation Reports) to ensure DQOs are met
Ted Johnson	SSHO	EIP	Develops and administers the site safety and health plan Manages personnel and environmental monitoring Coordinates preparation of job safety analyses Selects appropriate personal protective equipment and facilitates daily safety meetings Reviews essential H&S requirements with on-site personnel
Jackson Kiker	Program Chemist	EIP	Reviews the SAP Guides the selection of subcontract analytical laboratories Conducts field and laboratory audits or reviews audit results of designated personnel Serves as a POC for the Navy QAO Develops corrective action as required Serves as a technical advisor to the project

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Name	Title/Role	Organizational Affiliation	Responsibility
Kane McNeill	Project Chemist	EIP	Develops the project quality objectives and prepares the SAP. Selects qualified subcontract laboratories. Reviews radiological laboratory data on a routine basis. Informs Program Chemist and of Unusable/Rejected Data. Implements chemical data QC procedures and evaluates field performance. Reviews and approves validated laboratory data prior to use. Prepares the appropriate sections of the report summarizing the project sampling activities. Prepares, or designates personnel, to perform interim data quality assessment reports (for internal use only) to confirm DQO's are on track to meet the end of project goals. Makes necessary adjustments, with approval of Program chemist, if goals are not on track.
TBD	Data Manager	EIP	Stores, or confirms, all project records (hard copy and electronic) of field activities involving data recording, equipment calibration, and maintenance have been stored to the project shared drive folder. Works with Project Chemist to confirm all data provided from the lab (electronic lab reports and EDD's) and sample collection forms (i.e., COCs) are in electronic format on the project shared drive. Converts hardcopy data to electronic format (PDF's) when required. NIRIS: Confirms all data required for completing the required NIRIS uploads is available, complete, and up-to-date on weekly basis; conducts NIRIS uploads of non-laboratory data or designates project personnel to perform this function Confirms all data required to complete Data Quality Assessment Report has been collected and available in a format that allows for calculable values.
Rad Chem Field Technician(s)	Field Technician	Perma-Fix	Performs sampling in accordance with approved SAP Ensures that field QC samples are collected as specified in the SAP Completes field documentation and Implements field corrective actions as required Must have Occupational Safety and Health Administration (OSHA) 40-hour Certification and 8-hour OSHA Refresher Certification

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Name	Title/Role	Organizational Affiliation	Responsibility
Rhonda Ridenhower Nick Ly	Laboratory PM	Eurofins Test America, St Louis, MO. NVL Labs (Asbestos only)	Overseeing proper analysis and reporting of project samples according to approved SAP Managing communication between laboratory and EIP Project Chemist Ensuring proper QA/QC procedures are followed during laboratory analysis
Christina Rink-Ashdown	Data Validation PM	Laboratory Data Consultants	Performing data validation according to applicable methods and approved SAP Reviewing laboratory reports for compliance with applicable methods and approved SAP Applying validation qualifiers to analytical data Preparing data validation report NIRIS Upload of validated data

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SAP WS #8: SPECIAL PERSONNEL TRAINING REQUIREMENTS TABLE

Field personnel will be required to have completed OSHA 40-hour Hazardous Waste Operations and Emergency Response Standard Protection training, continued 8-hour Hazardous Waste Operations and Emergency Response, and submit to annual medical surveillance, as required by OSHA. The EIP SSHO will be responsible for ensuring that training and/or certification is met and that qualified personnel are performing the work.

Project Function	Specialized Training—Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records and Certificates
On-site project personnel	U.S. Department of Transportation/U.S. Nuclear Regulatory Commission 79-19 Regulatory Compliance Training	RSO / LSRO	Prior to start of fieldwork, if needed	On-site project personnel	Project Radiation Safety Officer or designee	Onsite and/or Project SharePoint
	Title 49 Transportation Subpart H Function-specific Training	RSO / LSRO	Prior to start of fieldwork	On-site project personnel	Radiological Personnel (RSO, ROM)	Onsite and /or Project SharePoint
	Radiation Worker Training (see Radiation Protection Plan, Parcel E, Hunters Point Naval Shipyard, San Francisco, California [EIP, 2019])	RSO / LSRO	Prior to start of fieldwork	On-site project personnel	SSHO	On-site and/or Project SharePoint
Rad Supervisor; Rad Tech; Rad Worker	Site-Specific Qualification as described in the "Parcel E Radiation Protection Plan, Section 3.5."	RSO / LSRO	Prior to start of fieldwork	Rad Supervisor; Rad Tech; Rad Worker	Radiological Personnel (LRSO, ROM)	Onsite and/or Project SharePoint

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SAP WS #9: PROJECT SCOPING SESSION PARTICIPANTS SHEET

Project Name: Radiological Confirmation Sampling and Survey at Parcel E	**Site Name:** Hunters Point, California, Parcel E
Projected Date(s) of Sampling: POP, September 16, 2019 – September 29, 2024	
PM: Matt Long (ECC Insights Philotechnics (EIP)	**Site Location:** Hunter Point, San Francisco, California
Date of Session: 08-October-2018 / 14:00	
Scoping Session Purpose: Kick Off Meeting / Teleconference	

Name	Title	Affiliation	Phone #	Email Address	Project Role
Paul Stoick	Lead RPM	NAVFAC	619-524-5755	paul.stoick@navy.mil	COR
Sean Ryan McCray	RPM	NAVFAC	619-507-2949	Sean-ryan.mccray.ctr@navy.mil	RPM
Hamid Naimi	QA	NAVFAC	510-501-1170	hamid.naimi@navy.mil	ROICC
Shirley Ng	H&S	NAVFAC	510-501-1170	shirley.ng@navy,mil	ROICC
Matt Long	Project Manager	ECC	774-244-7102	mlong@ecc.net	Project Manager
Marc Mizrahi	Program Manager	ECC	-	mmizrahi@ecc.net-	-
John Fern	Program Director	ECC	-	jfern@ecc.net-	-
August Ochabauer	VP Operations	ECC	-	aochabauer@ecc.net	-
Chris Peck	Project Engineer	ECC	-	cpeck@ecc.net	-
Eric Lanning	Project Manager	EIP	-	elanning@perma-fix.com	-
Alex Lopez	RSO, Tech Lead	EIP	970-778-0449	alopez@perma-fix.com	RSO

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	Review of Site Health and Safety • (EIP) – integrating with contractors on site at meeting next week Site Access and Security • (EIP) – any documents need to be completed prior to site access? • (NAVFAC) – Doug (NAVFAC) will manage any necessary documents for badging process. Communications • (EIP) – how can EIP support NAVFAC and other contractors with communications? • (NAVFAC) – primary vehicle for communications are contractor integration meetings (bi-yearly) to discuss logistics, concerns, frequency of meetings. • (NAVFAC) – meeting next week is strictly for Parcel E and will be good opportunity to discuss needs. Contract Administration • (EIP) – currently have executed contract and provided bonds. Anything else needed? • (NAVFAC) – all set, Paul S. (NAVFAC) will be COR. Draft Schedule Review • (EIP) – are we waiting on background soil report approval, or is the 21-day clock for draft schedule starting today? • (NAVFAC) – use background soil report for final revision, do not wait on it for the draft plan.
Mtg Minutes Notes	

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	Drawing native format files
	• (EIP) – requested editable (native) file formats in addition to PDF's to incorporate into plans.
	• (NAVFAC) – does EIP have access to Parcel G addendum and work plan?
	  • (NAVFAC) – should have been included in package. Will double check.
	  • (EIP) – Will also check.
	
	Action Items
	• EIP and NAVFAC to check if Parcel G addendum was provided.
	
	Next Scheduled Meeting
	• Contractor integration meeting next Thursday.

SAP WS #10: PROBLEM DEFINITION

Prior to the completion of Dec 2017 Radiological Data Evaluation Findings Report for Parcel E, investigation and removal actions, there were allegations that Tetra Tech EC, Inc. (TtEC) potentially manipulated and falsely represented data, and some allegations have since been confirmed. In addition, the on-site laboratory used a screening method to analyze radium-226 (^{226}Ra) that may have reported at levels higher than actual radioactivity. TtEC presented conceptual site models in remedial action completion reports (RACRs) that were based on potentially falsified data and screening results for ^{226}Ra reported by the on-site laboratory (results were biased high).

As a result, the Navy will conduct quality assurance confirmation investigations at radiologically impacted soil and the buildings in Parcel E that were previously surveyed by TtEC. The results from additional confirmation investigation activities to be performed as part pf this SAP and associated WP will be used to update the conceptual site model as needed. The goal of this work is to demonstrate that the unrestricted radiological free release letters for the former sanitary sewer (SS) and storm drain (SD) lines in Parcel E may be reinstated based on the evaluation of the quality assurance data to be collected as part of this SAP.

The planning team consists of the representatives of the Navy and EIP, LLC, with primary oversight by EPA and input from DTSC, CDPH, and Water Board. The lead agency at HPNS is the Navy and the lead federal regulatory agency is EPA. The Navy will continue to work with EPA and the State of California throughout the planning and site confirmation investigation process.

10.1 SITE LOCATION AND DESCRIPTION

HPNS is located in southeastern San Francisco on a peninsula that extends east into the San Francisco Bay. HPNS consists of 853 acres: 407 acres on land and 446 acres underwater. Parcel E consists of approximately 128 acres in the southwest portion of the HPNS and includes 17 existing buildings, 25 former buildings, and 1 ship berth. Historically, most of Parcel E was used as an industrial support area, which included a warehouse (Building 406) and the Former Oily Waste Ponds, where waste oil was stored until 1974. The Shoreline areas at Parcel E were used to store construction and industrial materials, as well as to dispose of industrial waste and construction debris. The NRDL used several Parcel E buildings during the 1950s and 1960s. The radiological areas of impact were identified in Figure 4 of the ROD for Parcel E (Dec-2013). **Figure 1** shows Parcel E in relation to the HPNS and the city of San Francisco, California.

10.2 SITE HISTORY

In 1939, the Navy purchased the land portion of HPNS and leased it to Bethlehem Steel Corporation. At the start of World War II in 1941, the Navy took possession of the property and operated it as a shipbuilding, repair, and maintenance facility until 1974 when the Navy deactivated HPNS. HPNS was also the site of the Naval Radiological Defense Laboratory from the late-1940s until 1969. From 1976 to 1986, the Navy leased HPNS to Triple A Machine Shop, Inc., a private ship repair company. In 1986, Triple A Machine Shop, Inc. ceased operations and the Navy resumed occupancy through 1989. In 1991, HPNS was placed on the Navy BRAC list, and its mission as a shipyard ended in April 1994. A history of Navy radiological operations at HPNS is provided in the *Final Historical Radiological Assessment, History of the Use of General Radioactive Materials, 1939–2003, Hunters Point Shipyard* (HRA; NAVSEA, 2004). The HRA concluded that low levels of radiological contamination exist within the confines of HPNS. The review of previous radiological activities, cleanup actions, and release surveys identified no imminent threat or substantial risk to tenants or the environment of HPNS or the local community.

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The SS/SD system was originally designed and built in the 1940s as a combined system to discharge into San Francisco Bay. The system was separated into SSs and SDs in 1958, 1973, and 1976, but was never fully completed.

The historical waste disposal activities at IR-02 and IR-03 included radio-luminescent commodity items such as dials, gauges, and deck markers, in conjunction with the disposal of other construction debris and industrial-wastes.

10.3 NATURE AND EXTENT OF POTENTIAL CONTAMINATION

The nature and extent of contamination at HPNS is described in ROD (Navy, 2013). The nature and extent of soils to be re-tested as part of the quality assurance activities of the is SAP is presented in the associated work plan. The potentially contaminated soils are associated with the former building's sites, storm drains, and storm sewers (**Figure 2**).

The two general radioactive sources of contamination were identified: the NRDL research facilities and historical waste disposal along the shoreline (identified as IR-02 and IR-03).

The NRDL research was performed primarily within the 500 series buildings and the building 707 triangle area. The activities performed at these buildings may have discharged small amounts of low-level radioactive liquids into the sanitary sewer, storm drain, and septic sewer lines; as a result, the sanitary sewer, storm drain, and septic sewer lines throughout Parcel E may have been radiologically impacted.

10.3.1 Radionuclides of Concern

Per the FINAL ROD for Parcel E (Navy, 2013), two general radioactive sources of contamination were identified: the NRDL research facilities and historical waste disposal along the shoreline (identified as IR-02 and IR-03).

The ROCs for the Parcel E Radiological Confirmation study are identified are as:

Cesium 137	Radium 226	Strontium 90	Plutonium 239	Uranium 235	Americium 241	Cobalt 60	Tritium
^{137}Cs	^{226}Ra	^{90}Sr	^{239}Pu	^{235}U	^{241}Am	^{60}Co	^{3}H

The NRDL research was performed primarily within the 500 series buildings and the Building 707 triangle area. The activities performed at these buildings may have discharged small amounts of low-level radioactive liquids into the sanitary sewer, storm drain, and septic sewer lines; as a result, the sanitary sewer, storm drain, and septic sewer lines throughout Parcel E may have been radiologically impacted.

The historical waste disposal activities at IR-02 and IR-03 included radio-luminescent commodity items such as dials, gauges, and deck markers, in conjunction with the disposal of other construction debris and industrial-wastes.

10.3.2 Potential Migration and Exposure Pathways

Potential migration pathways include:

- Releases to soil and air
- Releases to SS lines

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- Releases to SDs
- Releases from potentially leaking SS/SD lines to surrounding soil
- Release of sediments from breaks or seams during power washing of drain lines

Potential exposure pathways include:

- Soil:
  - External radiation from ROCs
  - Incidental ingestion and inhalation of soil and dust with ROCs for intrusive activities disturbing soil beneath the durable cover (only construction worker receptor)
- Building surfaces:
  - External radiation from ROCs
  - Worker Inhalation and incidental ingestion of re-suspended radionuclides

10.4 CURRENT STATUS

HPNS is not an active military installation and was selected for closure in 1991 pursuant to the terms of the Defense BRAC Act of 1990. The Navy leased many HPNS buildings to private tenants for more than 20 years. Parcels A, D-2, UC-1, and UC-2 have been transferred to the City and County of San Francisco for nondefense use, and the remaining areas of HPNS are planned to also be transferred. The known sources of contamination were removed by the Navy using the applicable standards at the time.

10.5 CONCEPTUAL SITE MODEL

Following previous investigation and removal actions, there were allegations that TtEC potentially manipulated and falsely represented data, and some allegations have since been confirmed. In addition, the onsite laboratory used a screening method to analyze radium-226 (^{226}Ra) that may have reported at levels higher than actual radioactivity. TtEC presented CSMs in remedial action completion reports (RACRs) that were based on potentially falsified data and screening results for ^{226}Ra reported by the onsite laboratory (results were biased high).

As a result, the Navy will conduct additional radiological investigations at the Parcel E soil and buildings that were previously surveyed by TtEC. The results of the additional investigation activities presented in this SAP and Work Plan will be used to update the CSM as needed.

Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)
Site Location	Located on San Francisco Bay near the southeastern boundary of San Francisco, California. HPNS encompasses approximately 848 acres, including approximately 416 acres on land, at the point of a high, rocky, 2-mile-long peninsula projecting southeastward into San Francisco Bay. Parcel E occupies approximately 128 acres of shoreline and lowland coast along the southwestern portion of HPNS (**Figure 1**).

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Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)
Site Operations and History	• Navy Radiological Defense Laboratory (NRDL) activities associated with analyzing samples from nuclear weapons tests, scientific studies (fallout, plant, animal, materials), and production and use of calibration sources. • The HRA also documents in Table 5-1 that the Navy had five radioactive licenses with the Atomic Energy Commission for ^{137}Cs, one for a quantity of 3,000 curies and a separate quantity of 20 curies of ^{137}Cs. Two licenses indicate that ^{137}Cs was in sources. In some cases, the Navy made its own sources with ^{137}Cs. • Use of radiography sources. • Use and potential disposal of radiological commodities, including discrete devices removed from ships (deck markers, radium dials) and welding rods. • Historical radiological material use documented in the HRA (NAVSEA, 2004) lists "impacted sites" – sites with potential for radioactive contamination. • Former surface soil impacted by fallout may be subsurface soil today because of fill activities.
Historical Site Conditions	Facility created from fill with some background levels of radionuclides (e.g., NORM and fallout). Dredge spoils from local berths were used as fill for some areas. Trenches were backfilled following removal of sewer lines. Trench backfill is mixed, but documentation of source is available (onsite fill, offsite fill, or mixture). Bay mud or bedrock marks bottom extent of fill material. Site drainage system was designed in the 1940s to discharge to San Francisco Bay and was separated into sanitary sewers and storm drains in 1958, 1973, and 1976, but never completed.

Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)	
Potential Source Areas	**Potential Historical Sources of Radiological Contamination**	• Potential spills and releases from the following: – Storage of samples from nuclear weapons tests at various NRDL facilities – NRDL waste disposal operations: ▪ Building 707 Triangle Area was used as NRDL waste receiving, packaging, and storage area ▪ Animal research at Building 707 • Incidental disposal of radioluminescent commodities (e.g., dials, deck markers) during maintenance, individually or attached to equipment. • Leaking radiography and calibration sources could affect buildings listed in HRA Table 6-1 related to production and maintenance of calibration sources. • Small amounts of low-level radioactive liquid waste were authorized for release with dilution to sanitary sewers based on regulations in place at the time.
	Release Areas in Parcel E	**Known Release Areas (from Section 6.4 of the HRA):** • Building 707 and Building 707 Triangle Area – Contaminated drainpipe in Building 707 – Contamination under concrete pad at Building 707 was remediated **Potential Releases Identified after the HRA:** • Unknown

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Impacted Buildings in Parcel E	**Impacted Buildings with High Contamination Potential (from Table 8-2 of HRA):** • Building 529 (demolished) – Measured 17 feet by 29.5 feet and included an underground isotope storage vault and waste sump. Building 529 was used as NRDL Isotope Storage Facility (Vault) and Neutron Generator. When the building was renovated for installation of the neutron generator, the vault was filled with compacted sand and capped with 8 inches of concrete. • Building 707 and Kennels - Building 707 is irregular in construction and measures approximately 13,000 square feet, including kennels. Building 707 was used as a research animal facility used by NRDL for animal breeding and housing, Waste Processing and Storage Facility, formerly leased as an animal clinic • Building 707 Triangle Area – This site is triangular in shape and measures approximately 223,000 square feet. This location is bounded by "J" Street, "I" Street, and 6th Avenue. The site was formerly used as NRDL radioactive waste receiving, packaging, and storage area and suspected septic tank and leach field from early operations. • Parcel E Shoreline? **Impacted Buildings with Moderate Contamination Potential (from Table 8-2 of HRA):** • Building 406 Building 406 is a common wood-framed, flush weather-boarded warehouse and shop-type building from WW II. The building is approximately 40,000 square feet and includes a central light and ventilation monitor joined by shallow shed roofs at either side. Building 406 was previously used as shipping, packing, and preserving and leased to an auto bodywork and towing company. • Building 500 Series – Consisting of demolished buildings and open space, the area of HPS where the 500 series buildings were located and is the location of the original RADLAB/NRDL facilities. In addition to the 500-series buildings, temporary structures were constructed, used, and removed, and radioactive materials were transported and stored outdoors. There were drain lines in buildings in the area, a dust collection system was constructed outside Building 506 for use by NRDL. Buildings 506, 507, 508, 510, 517, 520, 701 were vacated by NRDL when it moved to Building 815 in 1955. • Building 506 (demolished) - Irregular in construction and measured 9,375 square feet at the foundation. Building 506 was previously used to store radioactive waste containers stored on pad behind the building, NRDL biology and health physics laboratories, animal, nuclear and physical chemistry laboratories, NRDL chemistry laboratories, radiochemistry laboratory, NRDL instrument repair, darkroom, and densitometer for film badges, counting rooms, electro-physical and surface chemistry laboratories, administrative offices, storerooms, duty watch berthing, personnel decontamination, and RADLAB/NRDL headquarters and main facility prior to move to Building 815 in 1955. • Building 507 (demolished) - The original building measured approximately 35 feet by 190 feet and appears to have been constructed as a wooden-framed, corrugated building. Building 507 was used as the NRDL biology laboratories, NRDL change house and animal quarters, radiological decontamination center, biochemistry branch, physiology-psychology branch, and experimental pathology branch. • Building 508 (demolished) - The original Building 508 measured approximately 35 feet by 190 feet. Building 508 was used as the

Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)
	NRDL chemistry branch, library, personnel branch, photographic section of publications branch, radiological safety branch, barracks, health services division, chemical technology and nucleonics divisions, security division, health physics division, employee relations branch, and pathology laboratory. • Building 510 (demolished) - The original building measured approximately 30 feet by 85 feet. Building 510 was used as weapons test sample storage, non-NRDL training facility, NRDL radiation facility, glassblowing, woodworking and machine shops, physics branch, nuclear radiation branch, and research engineering section physics branch. • Building 517 (demolished) - The original building measured approximately 50 feet by 50 feet. Building 517 was used as the former brig and NRDL cobalt animal irradiation facility. • Building 520 (demolished) - The original building was irregular in construction and measured approximately 7,040 square feet at its foundation. Building 520 was used as the shipyard dental clinic and NRDL administrative offices. • Building 704 - Building 704 is a metal-sheathed shop building with a radioactive material storage area south of the building and adjacent to the animal pens/kennels. • Building 810 - Wood-framed structure, sided in flush weatherboard and includes a flat roof with shallow shed-roofed elements on all sides. The monitor includes vents but no windows. Building 810 was formerly used as LLRW and IDW storage location, storehouse, paint shop, and paint and oil storage. • Shack 79 (demolished) - The type of construction is unknown but thought to be wood. Dimensions were not available, and the shack was located behind Building 506. Shack 79 was formerly used for NRDL support for radioactive material. • Shack 80 (demolished) - The type of construction is unknown but thought to be wood. The shack was located behind Building 506. Shack 80 was formerly used for NRDL Support and was reported to have been moved from behind Building 506 to Building 704 area for "lab operations". **Impacted Buildings with Low or No Contamination Potential (from Table 8-2 of HRA):** • Building 414 - Wood-framed, corrugated metal-sided, 172-by-243-foot structure in the southern shipyard area that includes a tall, gabled main shop area with a shed-roofed extension to one side. Building 414 was formerly used as LLRW storage area for RI IDW, public works/supply storehouse, and Shaw Environmental & Infrastructure, Inc. site offices. • Building 521 - Building 521 is constructed of concrete and measures approximately 7,040 square feet at its foundation. Building 521 was used as a power plant and is one of two suspected sites of fuel oil burning from three OPERATION CROSSROADS target ships. • Building 701 (demolished) - The original building measured approximately 50 feet by 110 feet. Building 701 was used as a storage building that NRDL requested for "temporary" (120 days) storage of samples in 1947; however, the building was still in use by NRDL in

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Table 10-1. Conceptual Site Model

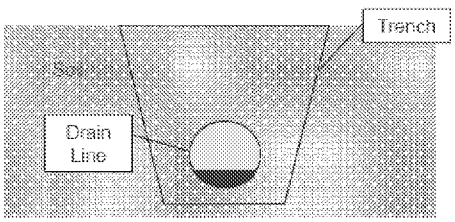
Site Name	Former Hunters Point Naval Shipyard (Parcel E)
	1954. • Building 704/Pens - Two former animal pens were identified on a 1949 map adjacent to the radioactive material storage area south of Building 704. The animal pens measured approximately 18 feet by 20 feet and 20 feet by 12 feet and have been removed. • Building 707 B (demolished) - This building is thought to have been of the same type as Building 708 (707A), a 50-foot by 20-foot Quonset hut. Building 707 B was used as the NRDL animal colony. • Building 707 C (demolished) - This building is thought to have been of the same type as Building 708 (707A), a 50-foot by 20-foot Quonset hut. Building 707 C was used as nuclear weapons test support and experimentation and equipment issue and receiving area. • Building 708 - Quonset hut adjacent to Building 707. It measures approximately 50 feet by 20 feet. Building 708 was formerly used as research animal facility, bio-medical facility, and animal psychology facility. • Building 807 (demolished) - This building measured approximately 60 feet by 28 feet. Building 807 was formerly used as scrap yard processing shed and possibly received scrap materials from ship decontamination.
Radionuclides of Concern for Parcel E (from Table 8-2 of HRA)[3]	• ^{226}Ra • ^{137}Cs • ^{90}Sr (for soil, former building sites, and interior surfaces of Buildings 521 and 810) • ^{3}H (only for former Building 506 site) • ^{239}Pu (only for interior surfaces of Building 521 and the sites of former Buildings 506, 510/510A, 704, 704 Storage Yard, 707 Triangle, and the 500 Series) • ^{60}Co (only for former Building 517 site) • ^{241}Am (only for former Buildings 506 and the 500 Series) • ^{235}U (only for former Building 707 Triangle site) • ^{239}Pu (only for interior surfaces of Building 521 and former Buildings 364 and 365)
Potential Migration Pathways	• Releases to soil and air. • Releases to sanitary sewer lines. – Buildings with known releases • Releases to storm drains. – Incomplete separation from sanitary sewer lines  Conceptual Cross Section of Drain Lines • Runoff from surface spills. • Releases from potentially leaking storm drain and sanitary sewer lines to surrounding soil (now removed). • Release of sediments from breaks or seams during power washing of drain lines.

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Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)
Potential Exposure Pathways	• Soil: – External radiation from ROCs – Incidental ingestion and inhalation of soil and dust with ROCs for intrusive activities disturbing soil beneath the durable cover (only construction worker receptor) • Building surfaces: – External radiation from ROCs – Inhalation and incidental ingestion of resuspended radionuclides
Current Status	• HPNS is not an active military installation. In 1991, HPNS was selected for closure pursuant to the terms of the Defense BRAC Act of 1990. For more than 20 years, the Navy leased many HPNS buildings to private tenants and Navy-related entities for industrial and artistic uses. Current leases include art studios and a police department facility. Parcels A, D-2, UC-1, and UC-2 have been transferred to the City and County of San Francisco for nondefense use, and the remaining areas of HPNS are also planned to be transferred. • All known sources removed by Navy using standards at the time. – Follow-up investigations resulted in removal of small volumes of soil to meet current RGs • Sanitary sewer and storm drain removal investigation conducted at Parcel E from 2010 to 2016. – Approximately 24,000 Linear Feet of trench line soil has been investigated and disposed of or cleared for use as onsite fill – Trench excavations that have been backfilled now contain homogenized soil from onsite fill, offsite fill, or a mixture of both

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Table 10-1. Conceptual Site Model

Site Name	Former Hunters Point Naval Shipyard (Parcel E)
Uncertainties	• Lower potential for radiological contamination than originally described in historical CSMs based on the following lines of evidence: – Known sources have been removed. – Sanitary sewers and storm drains, and 1 foot of soil surrounding the pipe removed to the extent practicable. The sewer lines were removed to within 10 feet of all buildings. Impacted buildings had remaining lines removed during surveys of the buildings. Non-impacted buildings had surveys performed at ends of pipes, and pipes were capped. – Any residual concentrations may be modified by radiological decay (shorter-lived radionuclides, such as ^{137}Cs and ^{90}Sr) or remobilization (including weathering and migration). – Overestimate of ^{226}Ra concentrations in soil by the onsite laboratory using an imprecise measurement method. • Data manipulation or falsification. • Data quality deficiencies. • ^{137}Cs and ^{90}Sr are present at HPNS because of global fallout from nuclear testing or accidents, in addition to being potentially present as a result of Navy activities. Because of backfill activities, ^{137}Cs and ^{90}Sr from fallout and Navy activities are not necessarily only on the surface and may be present in both surface and subsurface soil. • Potential for isolated radiological commodities randomly distributed around the site. • Trenches where scan data exceeded the IL and biased soil samples were not collected.

Notes:

^{241}Am = americium-241
^{60}Co = cobalt-60
^{137}Cs = cesium-137
^{3}H = hydrogen-3 / Tritium
^{226}Ra = radium-226
^{239}Pu = plutonium-239
^{90}Sr = strontium-90
^{235}U = uranium-235
BRAC = Base Realignment and Closure
IL = investigation level
LLRW = low-level radioactive waste
NORM = naturally occurring radioactive material
NRDL = Navy Radiological Defense Laboratory
pCi/g = picocuries per gram

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SAP WS #11: PROJECT QUALITY OBJECTIVES

Step	Project Quality Objectives
Step 1	**Define the Problem that Necessitates the Study**
Data manipulation and falsification were committed by a former contractor during the past SS and SD soil removal actions in Parcel E. The findings call into question the reliability of soil and building data, and there is uncertainty whether radiological contamination was present or remains in place. Therefore, the property is unable to be transferred as planned. Based on the uncertainty and the description of radiological activities in the HRA (NAVSEA, 2004), there is a potential for residual radioactivity to be present in soil and on building interior surfaces.	
Step 2	**Identify the Goal of the Study**
To determine if current site conditions regarding soil in the trench units, soil in the survey units, and building surfaces are compliant with the Parcel E ROD RAOs (Navy, 2013).	
Step 3	**Identify Information Inputs**

Surface soil and subsurface soil analytical data for the applicable ROCs provided by an accredited off-site laboratory will be used to make project decision. The ROCs (and their associated methods of analysis) for the soil areas are shown in WSs#15.1 through #15.4.
 	
Soil samples will be analyzed/scanned by the following methods:
• Laboratory Analyses
  o Gamma Spectroscopy — EPA Method 901.1 or equivalent
  o Alpha Spectroscopy — DOE Method A-01-R
  o Gas flow proportional counting (GFPC) — EPA Method 905.0 or equivalent
• Field Equipment Scans
• Gamma Surface Scans
 	
Building Investigation:
• Alpha-beta static, alpha and beta scan, and alpha-beta swipe data collected by radiological survey instruments on buildings and reference area surfaces	

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Step 4	Define the Boundaries of the Study

The soil sampling study boundaries include:

- Phase 1 and Phase 2 trench units (TUs),
- former building sites, and
- existing buildings identified in WS#18.1, 18.2, 18.3, 18.4 (*as defined in the previous studies.*)

Soil Sampling - Horizontal extent

- The former trench units and survey units (former building sites) will be re-marked by surveyors based on the previously recorded survey points.
- In addition, the trench units will have an additional 6 inches on each side wall removed.

Soil Sampling - Vertical extent:

- The Phase 1 and Phase 2 TUs will be based on the previously recorded maximum vertical extent of excavation will based on the values listed on WS 18.1 and 18.2. Vertical extent is not applicable for the survey units.

Building Surveys:

- The boundaries of existing buildings will be performed within the building interior. **Figure 2** shows the locations.

There are no practical constraints to data collection at these sites. There are no sensitive species that inhabit the site. Normal weather patterns should not impact site activities. However, heavy rain may delay excavation and radiological screening activities. Refer to WS#16 for estimated duration of fieldwork.

Step 5	Develop the Analytic Approach

- If the former building sites survey units and trench unit soil confirmation and investigation results demonstrate that there are no exceedances determined from a point-by-point comparison with the statistically-based remediation goals (RGs) or background (whichever is higher) at agreed upon statistical confidence levels, or that residual ROC concentrations are naturally occurring radioactive material (NORM) or anthropogenic background, then a RACR will be developed.
- If the former building site survey units and trench unit soil confirmation investigation results demonstrate exceedances of the RGs determined from a point-by-point comparison with the statistically-based RGs or background (whichever is higher) at agreed upon statistical confidence levels and are not shown to be NORM or anthropogenic background, then the Navy RPM will be notified.
- If one Phase 1 TU does not meet the Parcel E ROD RAO, then Phase 2 TUs will be excavated.
- If all of the Phase 1 TUs meet the Parcel E ROD RAO, then excavation of Phase 2 TUs will not be initiated.
  Multiple lines of evidence will be evaluated to determine whether site conditions are consistent with NORM or anthropogenic background. The data evaluation may include, but is not limited to, population-to-population comparisons, use of a maximum likelihood estimate or background threshold value, graphical comparisons, and comparison against RG's and regional background levels (whichever is higher).
- If result is greater than the selected criteria (comparison performed against both the RG and cannot be attributed to NORM or anthropogenic background), then remediation may be performed prior to backfill.
- If ^{90}Sr or ^{137}Cs is detected above release criteria, then additional analysis for isotopic ^{238}Pu/^{239}Pu/^{240}Pu or other alpha emitters may be performed to confirm contamination (**Figure 3**). If ^{226}Ra alpha isotope concentrations exceed the RG, then the soil sample will be analyzed for ^{234}U, ^{238}U, and ^{226}Ra using alpha spectroscopy (**Figure 4**). If the concentrations of the radionuclides in the uranium natural decay series are consistent with the assumption of secular equilibrium, then the ^{226}Ra concentration is NORM and site conditions comply with the ROD RAO.

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- Investigations of the Phase 2 trenches will consist of a combination of gamma scan surveys and soil samples. Phase 2 of the investigation evaluates the remaining TUs (listed on **Table 17-3**). Soil samples shall be collected with a drilling or direct push rig. One soil boring will be located every 50 linear feet along the former trench sidewalls.
- Existing Building Survey: for every identified survey unit within the existing building, 14 Alpha-Beta measurements will be collected. In addition, a swipe is required for every grid that an alpha beta measurement was collected from (14 per survey unit).

See compliance decision diagrams presented in the Parcel E WP for both soil and buildings (**Figure 3-1** and **Figure 4-6, respectively**).

Step 6	**Specify Performance or Acceptance Criteria**

To limit uncertainty in obtained environmental data, criteria for the precision, accuracy, representativeness, completeness, and comparability parameters and limit of detections decision level concentration (DLC) for the chemicals of concern have been developed to meet the RGs for the remedial actions. Measurement errors will be controlled by using appropriate sampling and analytical methods, adhering to the DoD QSM 5.3 (2019), following established standard operating procedures (SOPs), and having third-party data validation to verify laboratory processes and establish data usability for project decisions. The field crews will review the SAP before sample collection to limit sample collection errors. The subcontract analytical laboratory will have a copy of the SAP and will adhere to DoD QSM 5.3 guidance to limit measurement errors. MQO's are presented in WS#12 and WS#28.

Step 7	**Plan the Design for Obtaining Data**

Data for each component of the study will be obtained through the following methods (soil confirmation investigation):

- For Phase 1, 100 percent of soil will be re-excavated and characterized at former trench locations associated with former SSs and SDs. Soil sampling and scanning the TUs identified as part of Phase 2 would increase the confidence that current site conditions comply with the RAOs. Evaluation of the results of Phase 1 may lead to re-excavation of Phase 2 TUs .
- Phase 1 TUs — The radiological investigation will be conducted on a targeted group of TUs (from 1 to 7 feet deep) associated with former SSs and SDs. The Phase 1 TUs will be investigated using gamma scan surveys and soil sampling as described in WSs#14 and #17.
- Phase 2 TUs — Gamma scan surveys, soil sampling, and scanning of soil cores will be conducted on the remaining TUs. The Phase 2 TUs will be investigated as described in WSs#14 and #17. Phase 2 will only be performed if a Phase 1 RAOs is not met for an associated TU.

The soil samples collected will be analyzed for the applicable ROCs by accredited offsite laboratories and the results will be evaluated as described in **Step 5**. The excavated soil from within each trench and over-excavation will be tracked separately. EIP has selected to use the radiological screening yard (RSY) pads method for screening of the excavated material, consistent with *Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)* guidance (EPA 402-R-97-016, 2000).

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SAP WS #12: MEASUREMENT PERFORMANCE CRITERIA TABLE—SOIL FIELD QC SAMPLES

QC Sample	Analytical Group	Frequency	Data Quality Indicators	Measurement Performance Criteria
Field Duplicate	Gamma Isotopes – EPA Method 901.1 Gas Flow Proportional Counting (GFPC) -EPA Method 905.0 Alpha Isotopes – DOE Method A-01-R	One per every 10 field samples collected.	Precision	Relative percent difference (RPD) less than (<) 25 percent (%) for concentrations greater than (>) 5X the DLC Concentration of reported analytes are > 5X the DLC in either sample and absolute difference < 3X DLC
Equipment Blank		Disposable sampling equipment will be used as much as possible. However, if non-disposable equipment is use; one per day of field sampling for decontaminated equipment.	Bias / Contamination	No target analytes detected > minimum detectable concentration (MDC)
Field Blank		If Equipment Blank collected; one per source water per sampling event.	Bias / Contamination	No target analytes detected > MDC
Split Sample[1]		Soil samples will be retained for possible CDPH confirmatory analysis until the final RACR is issued.	N/A	None

Notes: [1] May be collected if requested by other stakeholders (EPA or CDPH) and will be evaluated by the stakeholder. Measurement and performance criteria will be outlined in the stakeholder guidance documents.

N/A = not applicable

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SAP WS #13: SECONDARY DATA CRITERIA AND LIMITATIONS TABLE

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
RGs	*Final Basewide Radiological Removal Action, Action Memorandum—Revision 2006, Hunters Point Shipyard, San Francisco, California*, Navy, April 2006	Navy, RGs for soil and surfaces	To determine whether site conditions in soil and building surfaces are compliant with the RGs for Parcel E.	Results will be compared to RGs and background, whichever is higher.
Parcel E	Record of Decision for Parcel E HPNS (2013)	site figures	Data will be used to confirm the boundaries for TUs and survey units (SUs) included in the Soil Investigations.	Electronic versions of previous excavations areas (if available) will be used to locate and mark the boundaries of former TUs and SUs.
Parcel E	*Draft-Final Radiological Data Evaluation Findings Report for Parcel E (Feb 2019)*	-	Background Information on the former studies that were falsified	Data considered to highly biased for field lab. Other falsified data is considered biased.

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SAP WS #14: SUMMARY OF PROJECT TASKS

14.1 SCOPE OF WORK

The following sections define the scope of work to be performed on this project.

14.1.1 Trench Units

The scope of work associated with the trench units identified in Phase 1 (and Phase 2, if required) include sampling activities that may include the following:

- Locating, confirming, and marking soil TU and SU boundaries.
- Removal and survey of the durable cover of Phase 1 TUs and Phase 2 TUs.

- Movement of equipment and materials to the site. Equipment mobilized to the site will undergo baseline radioactivity surveys in accordance with the Parcel E WP. Surveys will include direct scans, static measurements, and swipe samples. Equipment that fails baseline surveying will be removed from the site.
  - Surface and subsurface soil investigation with former SS and SD lines (TUs).
  - Phase 1 trench excavation and radiological soil screening and sampling.
  - Phase 2 trench gamma surface scan, soil borings, scans of the soil borings, and soil sample collection.
  - Third-party data review and validation.
  - Upload validated data to Naval Installation Restoration Information Solutions (NIRIS).

14.1.2 Survey Units / Former Building Sites

- Locating, confirming, and marking soil SU boundaries.
- Movement of equipment and materials to the site. Equipment mobilized to the site will undergo baseline radioactivity surveys in accordance with the Parcel E WP. Surveys will include direct scans and soil samples. Equipment that fails baseline surveying will be removed from the site.
- Surface investigation with former SUs.
- Upload data to Naval Installation Restoration Information Solutions (NIRIS).

14.1.3 Existing Buildings

- Locating and identifying existing buildings locations.
- Movement of equipment and materials to the site. Equipment mobilized to the site will undergo baseline radioactivity surveys in accordance with the Parcel E WP. Task will include directs scans, static measurements, and swipe samples. Equipment that fails baseline surveying will be removed from the site.
- Upload data to NIRIS.

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14.2 SAMPLE COLLECTION

14.2.1 Hand Auger Soil Sampling

Shallow soil boring samples (0 – 0.5 ft depth for Phase 2) may be collected using hand auguring techniques. Samples collected with a hand auger and/or slide hammer will either be fitted with a stainless-steel liner (slide hammer) or comprised of stainless steel (hand auger):

1. Obtain decontaminated stainless sleeves, hand auger, and slide hammer.
2. Put on a new, clean, and chemical-resistant pair of disposable gloves.
3. Auger to the desired sampling depth.
   - Option use slide hammer with clean sleeve to collect sample.
4. Remove the liner from the drive-tube and expose soil for sample collection.
5. Samples will be collected from within the zone of interest into the appropriate sample containers using a disposable scoop (or equivalent).
6. Sample Removal -
   - Via slide hammer - Upon removal of the tube from the ground, cuttings in the upper end of the tubs are removed, and the upper and lower ends of the tube are sealed. The soil tube will be turned over to the project geologist and radiation technician for sample preparation, radiological surveys, and containerization.
   - Via hand auger - Upon removal from the ground, the soil will be placed in the designated sampling jar.
7. The geologist will log the soil to provide accurate and consistent descriptions of soil characteristics. Soil boring logs will be maintained.
8. If required, the sample for radiological analyses will be mixed in the field by breaking the sample into small pieces and removing gravel. The depth, recovery position, and scan measurement information should be correlated to each sample extracted from the core.
9. Label, package, and prepare the samples for shipment to the laboratory.

Sample locations will be marked for future surveying using a pin flag or equivalent next to the boring location, or survey locations will be collected with hand-held global positioning system device.

14.2.2 Shallow Soil Sampling

Shallow soil sampling procedures shall be used to characterize and radiologically clear the excavated backfill (ESU) and the excavated surrounding soils (SFU's) on the RSY pads_to facilitate the MARSSIM (EPA 402-R-97-016, 2000), characterization survey. The shallow soil samples submitted for analysis will be collected using the general sampling technique described as follows:

1. Obtain a new sealed (unused) disposable sampling scoop (or equivalent).
2. Put on a new (unused) pair of sampling gloves and other appropriate personal protective equipment.
3. For RSY pads and the former building sites, establish the systematic triangle grid pattern (visa the VSP program or equivalent) throughout designated area using a randomly selected starting point.

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4. For S3 (this option was selected by EIP), collect samples at timed interval equivalent to one per 11 cubic yards (approximately). The minimum number of samples required is 19 per SU (or 200-cubic-yard equivalent). Samples will be collected at timed interval equivalent to one soil sample every 10 minutes (approximately) as appropriate from the conveyor belt.
5. Refer to WS #19 for required sample volume(s). Collect twice the required volume if the sample location is selected as a field duplicate or split sample. Split samples will be available to USEPA or other regulatory agencies to take for independent analysis real-time during field activities. Locations for split samples will be flexible to allow for sufficient volume for Navy samples and Quality Assurance (QA)/QC samples.
6. A representative sample will be prepared for laboratory analysis. The sample for radiological analyses will be mixed in the field by breaking the sample into small pieces and removing overburden gravel and biological material (equivalent to approximately 3.5 to 4-mesh sieve size). Mixing by hand will be conducted until there is consistency in texture and color. The entire mixed sample, or aliquot thereof, will be placed in the designated laboratory sample container. For field duplicate samples (1 for every 10 field samples collected) or split samples, an alternate spooning technique will be used to split the soil between the primary sample container and the field duplicate or split sample container. Collect the soil samples for analysis into the appropriate sample containers using a disposable sampling scoop (or equivalent).
7. Soil samples will be submitted for gamma spectroscopy analysis. Up to 10% or more of the samples may also be analyzed for ^{90}Sr. Alpha analyses will be submitted as needed. Refer to Figures 3, 4, and 5 for details on selecting secondary analyses.
8. Label, package, and prepare the samples for shipment to the laboratory(s) or split sample submission to the laboratory(s) designated for the collected analysis per WS #27
9. Sample containers will be radiologically released from the radiological areas prior to shipment to the laboratory(s).

14.2.3 Direct Push Technique Soil Sampling

Phase 2 soil boring samples will be collected using direct push sampling techniques. The direct push drilling technique typically incorporates a 2-inch minimum diameter outer casing and inner drive-tube containing 1.5-inch diameter or equivalent acetate liners. Soil samples will be collected using the general sampling techniques described as follows:

1. Direct the drilling subcontractor or sampling crew to the desired location.
2. Advance the drive-tube (with acetate liner) into the subsurface driving soil into the acetate liner.
3. Put on a new, clean, and chemical-resistant pair of disposable gloves.
4. Remove the liner from the drive-tube and expose soil for sample collection.
5. Samples will be collected from within the zone of interest into the appropriate sample containers using a disposable scoop (or equivalent).
6. Upon removal of the tube from the ground, drill cuttings in the upper end of the tubs are removed, and the upper and lower ends of the tube are sealed. The soil tube will be turned over to the project geologist and radiation technician for sample preparation, radiological surveys, and

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containerization. Once retrieved from the hole, the tube is carefully cut open to maintain the material in the tube.

7. The geologist will log the soil boring to provide accurate and consistent descriptions of soil characteristics. Soil boring logs will be maintained.

8. 250-400 grams of soil (approximately 1 cup) is required to complete required analyses, 500 to 800 grams if the sample is selected as a field duplicate or split sample. If sample size requirements are not met by a single sample collection, then additional sample volume may be obtained by collecting a sample from below the original sample location within the core and compositing the sample.

9. The entire mixed sample will be placed in the designated laboratory sample container and the range of soil depths included in the sample recorded in the field logbook.

10. When a field duplicate sample is required (1 for every 10 field samples collected), the sample will be evenly split following mixing of the material and removal of extraneous material, and each aliquot placed into an appropriately labeled sample container. The sample for radiological analyses will be mixed in the field by breaking the sample into small pieces and removing gravel. The depth, recovery position, and scan measurement information should be correlated to each sample extracted from the core.

11. Label, package, and prepare the samples for shipment to the laboratory(s).

14.2.4 Swipe Sample

The surveys of the existing buildings will utilize this sample procedure.

1. Swipe sample size area is approximately 100 cm^2
2. On-site analysis equipment: Ludlum model 3030 or equivalent
   a. offsite analysis is not associated with the swipe samples.
3. Sampling equipment and materials
   a. Gloves
   b. 5.1 cm swipe
4. Procedure:
   a. Don new and unused approved sampling gloves
   b. Measure and mark out area (preferred grid size: 10 cm by 10 cm)
   c. Note the required sample details on the sampling form and the logbook
      1) Sample ID
      2) Sample Date and Time
      3) Sample Location
      4) Field Tech Collecting Sample
      5) CoC#
      6) Any deviations or issues with sample collection conditions
   d. Wipe in an "S" pattern –
      1) right to left or left to right on top of sample area,
      2) moving down 1 diameter of the swiped area that was covered,
      3) swipe in opposite direction,
      4) moving down 1 diameter of the swiped area that was covered,
      5) repeat until at the bottom of sample area

e. Place sample in approved device for analysis.
f. Properly dispose of sampling gloves and swipe in the designated IDW container.

14.2.5 Equipment Decontamination

Sampling equipment decontamination is not anticipated for this project since disposable equipment will be used as much as possible. However, if decontamination is necessary the following procedures will be used. Decontamination of non-disposable sampling equipment that comes in contact with samples (i.e., sleeve rings and the split-spoon sampling device) will be performed to prevent the introduction of extraneous material into samples, and to prevent cross contamination between samples. If required, sampling equipment will be decontaminated by steam cleaning or by washing with a non-phosphate detergent such as Liquinox™ or equivalent. Decontamination water will be collected in 55-gallon U.S. Department of Transportation-approved drums or a poly-tank.

The following procedures will be used for decontamination of non-disposable sampling equipment:

1. If mud or soil is adhering to the sampling equipment, first rinse with potable water. This step will decrease the gross contamination and reduce the frequency at which the non-phosphate detergent and water solution need to be changed. This process may include scrubbing or
2. Wash with the non-phosphate detergent and water solution. Dilute the non-phosphate detergent as directed by the manufacturer. This step will remove remaining contamination from the equipment.
3. Rinse with potable water.
4. Rinse with deionized water. This step will rinse detergent solution and potable water residues. Rinsing will be done by applying the deionized water from a clean squeeze bottle (or equivalent) while holding equipment over a bucket.
5. Dispose of decontamination water in designated IDW container for liquids.
6. Record Volume and associated site (Building number and SU, TU, etc.) on disposal log.

The WP discuses radiological scanning of decontaminated equipment.

Refer to the RPP for details on equipment calibration

14.3 CALIBRATION

Portable survey instruments will be calibrated annually at a minimum, in accordance with American National Standards Institute (ANSI) N323a-1997 Radiation Protection Instrumentation Test and Calibration, Portable Survey Instruments (ANSI N323) (ANSI, 1997), or an applicable later version. Instruments will be removed from service on or before calibration due dates for recalibration. If ANSI N323 does not provide a standard method, the calibration facility should comply with the manufacturer's recommended method.

14.4 DAILY PERFORMANCE CHECKS

Before use of the portable survey instruments, calibration verification, physical inspection, battery check, and source-response check will be performed in accordance with SOP RP-108, *Count Rate Instruments,* and SOP RP-109, *Dose Rate Instruments* (**Attachment 3**), or equivalent. Portable survey instruments will have a current calibration label that will be verified daily prior to use of the instrument.

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Physical inspection of the portable survey instrument will include the following:

- General physical condition of the instrument and detector before each use
- Knobs, buttons, cables, connectors
- Meter movements and displays
- Instrument cases
- Probe and probe windows
- Other physical properties that may affect the proper operation of the instrument or detector

Any portable survey instrument or detector having a questionable physical condition will not be used until problems have been corrected. A battery check will be performed to ensure that sufficient voltage is being supplied to the detector and instrument circuitry for proper operation. This check will be performed in accordance with the instrument's operations manual. The instrument will be exposed to the appropriate (alpha, beta, gamma) check source to verify that the instrument response is within the plus or minus 20 percent range determined during the initial response check. The calibration certificates and daily QA/QC records for each instrument used and the instrument setup test records will be provided in the project report.

If any portable survey instrument, or instrument and detector combination, having a questionable physical condition that cannot be corrected fails any of the operation checks stated in SOP RP-108, *Count Rate Instruments*, or SOP RP-109, *Dose Rate Instruments* (**Attachment 3**), or has exceeded its annual calibration date without RSO approval, then the instrument will be put in an "out of service" condition. This is done by placing an "out of service" tag or equivalent on the instrument and securing the instrument or the instrument and detector combination in a separate area such that the instrument and instrument and detector combination cannot be issued for use. The RSO and Radiological Control Technician (RCT) and their respective supervisors will be notified immediately when any survey instrumentation has been placed "out of service". Instruments tagged as "out of service" will not be returned to service until all deficiencies have been corrected. The results of the daily operation checks, previously discussed, will be documented.

14.5 Data Management

Radiological survey and environmental data will be maintained in accordance with SAP WS#29. Validated analytical data and field screening data will be uploaded into the Navy's centralized database (NIRIS) and will be included in or referenced in final reports.

14.5.1 Data Verification

An EIP Manager with knowledge of radiological QA/QC requirements will be present in the field for the duration of soil confirmation sampling activities. This manager's sole responsibility will be to ensure that the QC measures in the project plans are performed. The manager will maintain QA/QC records for review and provide copies in the final report.

The contractor will conduct weekly QC meetings to keep Navy personnel informed of field progress. The contractor will prepare meeting materials, including agenda, figures, data, and look-ahead calendar, and provide copies to participants 24 hours in advance of the meeting. Meeting minutes will be provided to the Navy within 48 hours of the meeting. Additional details regarding data verification are presented in WSs#34-36.

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14.5.2 Electronic Data Deliverables

The analytical laboratory(s) will generate and provide the Electronic Data Deliverables (EDDs) for all sample data packages analyzed. The EIP Project Chemist will work with the selected laboratory(s) to ensure their EDDs meet the NIRIS EDD (NEDD) requirements prior to generation. Upon completion of the data validation, the analytical results will be reviewed by the EIP Project Chemist, and either approved for use on the project or not approved for use.

For data that is not approved for use, the Project Chemist will inform the laboratory(s) or data validator of the issue. Once the documented issue has been resolved, the data will be re-submitted for review to the Project Chemist. The EDD will only be prepared for loading once the data has been approved for use. The project required NEDD applicable field data that is not present in the laboratory(s) provided EDD will be entered and/or loaded by the data manager or the project personnel designated by the PM.

At the completion of the project EIP, or the personnel designated by the PM, will generate a NEDD compliant file(s) from the EDMS, then load and submit the compliant files to the NIRIS.

14.5.3 Data Validation

Analytical data validation will be conducted by an independent third-party data validation subcontractor in accordance with WSs#34 through #36 and consistent with the following

- *General Data Validation Guidelines* (DoD, 2019)
- *Data Validation Guidelines, Module 1* (DoD, 2020)
- *Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP)* (MARLAP; EPA et al., 2004), and
- MARSSIM (EPA 402-R-97-016, 2000).

USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review, SFAM01.1 (EPA, 2020b) may also be applicable. The data validation findings are summarized in a project specific data validation report. The report content will include an introduction that includes validation guidance used, a summary of the QC elements reviewed, a description of deficiencies, and a summary of the data qualification.

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SAP WS #15: REFERENCE LIMITS AND EVALUATION TABLES

SAP WS #15.1: Reference Limits and Evaluation Table — Site Contaminants (Soil Matrix — Gamma Isotopes)

Analyte	CAS Number	Units	Project Action Limit / Remedial Goals	Project Action Limit Reference[5]	Project Quantitation Limit Goal	Laboratory-Specific Limits
						DLC[3]
Cesium-137 (^{137}Cs)	10045-97-3	pCi/g	0.141[1]	BTV [1]	0.070	0.070
Radium-226 (^{226}Ra)	13982-63-3	pCi/g	1.0 above background[2]	ROD	0.10	0.10
Cobalt-60 (^{60}Co)	10198-40-0	pCi/g	0.252[1]	ROD	0.1	0.1
Americium-241 (^{241}Am)	14596-10-2	pCi/g	1.36[1]	ROD	0.7	0.7

Notes:

[1] These values are provided in the Final Record of Decision for Parcel E, HPNS (Navy, 2013), with the exception of ^{137}Cs. The value for ^{137}Cs is the offsite background threshold value (BTV) determined in *Final Background Soil Study Report, Base Realignment and Closure Program Management Office West. Former Hunters Point Naval Shipyard, San Francisco, California* (CH2MHill, 2020).

[2] The ^{226}Ra background for definitive data is 0.861 pCi/g based on the offsite BTV determined in the *Final Background Soil Study Report, Base Realignment and Closure Program Management Office West. Former Hunters Point Naval Shipyard, San Francisco, California* (CH2MHill, 2020). If a new background dataset is collected and approved for Navy use, which may establish background concentration for ROCs, that background dataset may be used in place of the existing background concentration. The comparison will be performed against both the RG and background, using the higher of the two values for the final evaluation.

[3] DLC at or less than the value listed are goals for ^{137}Cs and ^{226}Ra for samples for this project. Project DLCs for radiological analyses are calculated on a sample specific basis and will vary. The values listed indicate a minimum DLC that will be achieved for the majority of samples, under normal analytical conditions. For samples reported as undetected (U-qualified), the DLC can be highly variable. For samples reported as not-detected with positive results, if the DLC exceeds the release criteria, the laboratory will be requested to recount the samples to allow re-evaluation of the reported sample results. DLC for other radionuclides analyzed by gamma spectroscopy are not required to be achieved unless specifically requested on the applicable chain of custody. Gamma spectroscopy analyses will be based on meeting the DLCs for ^{137}Cs and ^{226}Ra. DLCs for other radionuclides analyzed by gamma spectroscopy are not required to be achieved but will be reported by the laboratory.

[4] Soil Matrix-Gamma isotopes will be analyzed under EPA Method 901.1 or equivalent. The applicable laboratory SOP is referenced on WS#23.

[5] Final Record of Decision for Parcel E, HPNS (Navy, 2013) - Table 8: Remediation Goals for RadioNuclides

CAS = Chemical Abstract number
pCi/g = picocuries per gram

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SAP WS #15.2: Reference Limits and Evaluation Table (Soil Matrix — Alpha Isotopes)

If the gamma spectroscopy laboratory results indicate a concentration of ^{226}Ra above the RG in a sample, then the sample will be analyzed using alpha spectroscopy for ^{238}U, ^{234}U, and ^{226}Ra to evaluate equilibrium conditions (WS#11 / Step 5)(**Figure 3** and **Figure 4**).

Analyte	CAS Number	Units	Project Action Limit / Remedial Goals	Project Action Limit Reference[8]	Project Quantitation Limit Goal	Laboratory-Specific Limits[1,2]
						DLC
Radium-226 (^{226}Ra)[3]	13982-63-3	pCi/g	1.0 above background[4]	ROD	0.25	TBD
Americium-241 (^{241}Am)	14596-10-2	pCi/g	1.36 (residential)	ROD	0.1	TBD
Plutonium-238 (^{238}Pu)	13981-16-3	pCi/g	none	--	0.5	0.5
Plutonium-239/240 ($^{239/240}$Pu)[5]	13981-16-3	pCi/g	2.59 (residential)	ROD	0.5	0.5
Uranium-234 (^{234}U)	13966-29-5	pCi/g	none	--	0.5	0.5
Uranium-235/236 ($^{235/236}$U)[6]	15117-96-1	pCi/g	0.195 (residential)	ROD	0.1	0.1
Uranium-238 (^{238}U)	7440-61-1	pCi/g	none	--	0.5	0.5

Notes:

[1] Results for non-aqueous samples are reported on a dry-weight basis.

[2] Project DLCs for radiological analyses are calculated on a sample specific basis and will vary. The values listed indicate a minimum DLC that will be achieved for the majority of samples, under normal analytical conditions. For samples reported as undetected (U-qualified), the DLC can be highly variable. For samples reported as not-detected with positive results, if the DLC exceeds the release criteria, the laboratory will be requested to recount the samples to allow re-evaluation of the reported sample results. Gamma spectroscopy analyses will be based on meeting the DLCs for ^{137}Cs and ^{226}Ra. DLC for other radionuclides analyzed by alpha spectroscopy are not required to be achieved unless specifically requested on the applicable chain of custody.

[3] Where possible, isotopic analysis for ^{226}Ra will be performed using the same dissolution/digestion as ^{238}U to ensure comparability of results. Detected radium isotopes will be reported.

[4] Final Background Soil Study Report, Base Realignment and Closure Program Management Office West, Former Hunters Point Naval Shipyard, San Francisco, California (CH2M Hill, Inc., 2020), the initial background threshold value (BTV) to be used for site-specific data comparisons is the offsite value of 0.861 pCi/g. The ^{226}Ra RG is defined as 1 picocurie per gram (pCi/g) plus background. Plutonium is only analyzed if ^{90}Sr is detected.

[5] Plutonium-239 is listed in the above table as plutonium-239/240 because the alpha energy peaks for the isotope of plutonium cannot be separated in alpha spectroscopy. Therefore, the laboratory will report as listed above in the table. Detected plutonium isotopes will be reported. RGs listed on Table 8 of Parcel E ROD

[6] Uranium-235 is listed in the above table as uranium-235/236 because the alpha energy peaks for the isotope of uranium cannot be separated in alpha spectroscopy. Therefore, the laboratory will report as listed above in the table. Detected uranium isotopes will be reported.

[7] The Soil-Matrix-Alpha Isotopes will be analyzed under DOE method A-01-R. The applicable lab SOP is referenced on WS#23.

[8] Final Record of Decision for Parcel E, HPNS (Navy, 2013) - Table 8: Remediation Goals for RadioNuclides

CAS = Chemical Abstract Number
none = Not a radionuclide of concern for the site, but reported in the laboratory gamma spec standard list
pCi/g = picocuries per gram

SAP WS #15.3: Reference Limits and Evaluation Table — Site Contaminants (Soil Matrix — GFPC / Strontium-90)

Analyte	CAS Number	Units	Project Action Limit / Remedial Goals	Project Action Limit Reference	Project Quantitation Limit Goal (MDC)	Laboratory-Specific
						DLC
Strontium-90 (^{90}Sr)[1]	10098-97-2	pCi/g	0.331[2] (residential)	ROD	0.15	0.15

Notes:

[1] In addition to the samples analyzed for ^{90}Sr, if laboratory results indicate a concentration of ^{137}Cs above the RG in a sample, the sample will be analyzed for ^{90}Sr. Refer to Figure 5 for possible additional analyses.

[2] Value based on the remedial goals cited in the Final Record of Decision for Parcel E, HPNS (Navy, 2013). Comparison will be based on the RG or background, whichever is higher.

[3] The Soil Matrix -Strontium 90 will be prepared per SOP ST-RC-0058 and analyzed under EPA Method 905.0 or equivalent. The applicable lab SOPs are referenced on WS#23.

[4] Reference **Figure 4** for additional analyses that may be required if present above the RG.

CAS = Chemical Abstract Number
pCi/g = picocuries per gram

SAP WS #15.4: Reference Limits and Evaluation Table — Site Contaminants (Soil Matrix — Tritium [^3H])

Analyte[1]	CAS Number	Units	Project Action Limit / Remedial Goals	Project Action Limit Reference	Project Quantitation Limit Goal (MDC)	Laboratory-Specific[2]
						DLC
Tritium (^3H)	10028-17-8	pCi/g	N/A	N/A	-	-

Notes:

[1] Only associated with two Building Site Surveys – Buildings 506 and 529.

[2] Method of Analysis – EPA Method 905.0

N/A = Not Applicable. Tritium was not identified in Parcel E ROD (Navy, 2013); however, it was listed in the PWS as an ROC for the two buildings listed in note 1.

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SAP WS #15.5: Reference Limits and Evaluation Table — Soil Backfill

Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific		
					LOQ	LOD	MDL
		mg/kg					
Volatiles / 8260							
1,1,1,2-Tetrachloroethane	630-20-6	2	EPA RSL[1]	1	0.005	0.005	0.00222
1,1,1-Trichloroethane	71-55-6	8,100	EPA RSL	100	0.005	0.005	0.00198
1,1,2,2-Tetrachloroethane	79-34-5	0.6	EPA RSL	0.5	0.005	0.0008	0.000285
1,1,2-Trichloroethane	79-00-5	1.1	EPA RSL	0.5	0.005	0.0032	0.00088
1,1-Dichloroethane	75-34-3	3.6	EPA RSL	1	0.005	0.0008	0.00021
1,1-Dichloroethene	75-35-4	230	EPA RSL	100	0.005	0.0016	0.00059
1,2,3-Trichlorobenzene	87-61-6	63	EPA RSL	1	0.005	0.0032	0.00081
1,2,3-Trichloropropane	96-18-4	0.0051	EPA RSL	0.005	0.005	0.0008	0.000218
1,2,4-Trichlorobenzene	120-82-1	24	EPA RSL	1	0.005	0.0016	0.00073
1,2,4-Trimethylbenzene	95-63-6	300	EPA RSL	50	0.005	0.005	0.00231
1,2-Dibromo-3-chloropropane	96-12-8	0.0053	EPA RSL	0.005	0.01	0.01	0.00366
1,2-Dichlorobenzene	95-50-1	1,800	EPA RSL	100	0.005	0.005	0.00187
1,2-Dichloroethane	107-06-2	0.46	EPA RSL	0.2	0.005	0.0016	0.0007
1,2-Dichloropropane	78-87-5	2.5	EPA RSL	0.5	0.005	0.0016	0.00055
1,3,5-Trimethylbenzene	108-67-8	270	EPA RSL	50	0.005	0.005	0.00242
1,3-Dichloropropane	142-28-9	1,600	EPA RSL	100	0.005	0.0004	0.000173
1,4-Dichlorobenzene	106-46-7	2.6	EPA RSL	1	0.005	0.0008	0.000245
2-Butanone	78-93-3	27,000	EPA RSL	500	0.02	0.0128	0.00389
2-Chlorotoluene	95-49-8	1,600	EPA RSL	100	0.005	0.0016	0.00051
2-Hexanone	591-78-6	200	EPA RSL	100	0.02	0.0128	0.00489
4-Chlorotoluene	106-43-4	1,600	EPA RSL	80	0.005	0.0008	0.000361
4-Methyl-2-pentanone	108-10-1	33,000	EPA RSL	100	0.02	0.0128	0.00436
Acetone	67-64-1	61,000	EPA RSL	500	0.072	0.072	0.0356
Benzene	71-43-2	1.2	EPA RSL	0.5	0.005	0.0004	0.000151
Bromobenzene	108-86-1	290	EPA RSL	1	0.005	0.0016	0.00049
Bromochloromethane	74-97-5	150	EPA RSL	1	0.005	0.005	0.00246
Bromodichloromethane	75-27-4	0.29	EPA RSL	0.1	0.005	0.005	0.00213
Bromoform	75-25-2	19	EPA RSL	10	0.0051	0.0051	0.00255
Bromomethane	74-83-9	6.8	EPA RSL	1	0.01	0.0032	0.00135

Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific		
					LOQ	LOD	MDL
		mg/kg					
Volatiles / 8260							
Carbon Disulfide	75-15-0	770	EPA RSL	10	0.005	0.005	0.00166
Carbon Tetrachloride	56-23-5	0.65	EPA RSL	0.1	0.005	0.005	0.00201
Chlorobenzene	108-90-7	280	EPA RSL	10	0.005	0.005	0.00206
Chloroform	67-66-3	0.32	EPA RSL	0.5	0.01	0.0008	0.00029
Chloromethane	74-87-3	110	EPA RSL	10	0.01	0.0016	0.00077
cis-1,2-Dichloroethene	156-59-2	160	EPA RSL	10	0.005	0.0008	0.000201
Dibromochloromethane	124-48-1	8.3	EPA RSL	1	0.005	0.005	0.00227
Dibromomethane	74-95-3	24	EPA RSL	1	0.005	0.0008	0.000317
Dichlorodifluoromethane (Freon 12)	75-71-8	87	EPA RSL	10	0.01	0.0064	0.00274
Ethyl benzene	100-41-4	5.8	EPA RSL	2	0.005	0.0008	0.000305
Ethylene Dibromide	106-93-4	0.036	EPA RSL	0.01	0.005	0.005	0.00217
Hexachlorobutadiene	87-68-3	1.2	EPA RSL	1	0.02	0.0064	0.00211
Methyl tertiary butyl ether	1634-04-4	47	EPA RSL	20	0.005	0.0032	0.0016
Methylene Chloride	75-09-2	57	EPA RSL	5	0.0032	0.0032	0.00104
m/p-Xylene	108-38-3	550	EPA RSL	50	0.005	0.0016	0.00056
n-Butylbenzene	104-51-8	3,900	EPA RSL	10	0.005	0.0016	0.00058
Propyl benzene	103-65-1	3,800	EPA RSL	10			
o-Xylene	95-47-6	650	EPA RSL	50	0.005	0.0008	0.000266
Styrene	100-42-5	6,000	EPA RSL	100	0.005	0.0008	0.00028
Tetrachloroethene	127-18-4	24	EPA RSL	1	0.005	0.005	0.00191
Toluene	108-88-3	4,900	EPA RSL	100	0.005	0.0008	0.000227
trans-1,2-Dichloroethene	156-60-5	70	EPA RSL	100	0.005	0.0008	0.00039
trans-1,3-Dichloropropene	10061-02-6	1.8	EPA RSL	1	0.005	0.0002	0.000083
Trichloroethene	79-01-6	0.94	EPA RSL	0.1	0.005	0.005	0.00191
Trichlorofluoromethane	75-69-4	23,000	EPA RSL	10	0.01	0.01	0.0032
Vinyl Chloride	75-01-4	0.059	EPA RSL	0.04	0.005	0.0032	0.00134

Notes: [1] EPA regional screening level (RSL) for residential soil, April 2019, applies to all compounds with EPA RSL reference.

CAS = Chemical Abstracts Service
LOQ = Limit of Quantitation
LOD = Limit of Detection
MDL = Method Detection Limit

mg/kg = milligrams per kilogram

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Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific		
					LOQ	LOD	MDL
		mg/kg					
Metals / 6020							
Antimony	7440-36-0	31	EPA RSL[1]	10	2	1.5	0.733
Arsenic	7440-38-2	11.1	HPAL[2]	10	2.5	2	0.665
Barium	7440-39-3	15,000	EPA RSL	50	2	0.9	0.296
Beryllium	7440-41-7	160	EPA RSL	10	0.5	0.12	0.033
Cadmium	7440-43-9	71	EPA RSL	10	0.5	0.15	0.041
Chromium	7440-47-3	120,000	EPA RSL	50	3.5	0.4	0.123
Cobalt	7440-48-4	23	EPA RSL	10	1	0.2	0.0679
Copper	7440-50-8	3,100	EPA RSL	50	5	0.8	0.217
Lead	7439-92-1	80	*Human Health and Ecological Risk Office, HERO HHRA NOTE NUMBER: 3, DTSC-modified Screening Levels* (DTSC, 2019)	50	0.9	0.8	0.31
Manganese	7439-96-5	1,400	HPAL	50	4.5	0.4	0.1
Molybdenum	7439-98-7	390	EPA RSL	50	2.5	0.4	0.115
Nickel	7440-02-0	1,500	EPA RSL	50	4	0.45	0.132
Selenium	7782-49-2	390	EPA RSL	50	3	1.75	0.86
Silver	7440-22-4	390	EPA RSL	50	1.5	0.6	0.16
Thallium[3]	7440-28-0	0.81 / 0.78	HPAL / EPA RSL	0.65	3	1.3	0.65
Vanadium	7440-62-2	390	EPA RSL	50	2	0.35	0.094
Zinc	7440-66-6	23,000	EPA RSL	*50*	8	1.5	0.398
Metals / 7471							
Mercury	7439-97-6	11	EPA RSL	10	0.017	0.0133	0.00553

Notes: [1] EPA RSL for residential soil, April 2021, applies to all compounds with EPA RSL reference.
[2] Calculation of Hunters Point Ambient Levels (HPAL) (PRC Environmental Management Inc., 1995).
[3] Thallium will be evaluated using the laboratory RL.
CAS = Chemical Abstracts Service
LOQ = Limit of Quantitation
LOD = Limit of Detection
MDL = Method Detection Limit mg/kg = milligrams per kilogram

Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific Limit		
					LOQ	LOD	MDL
		mg/kg					
Polychlorinated Biphenyls (PCB's) / 8082							
Aroclor-1016	12674-11-2	4.1	EPA RSL[1]	2	0.066	0.0307	0.0102
Aroclor-1221	11104-28-2	0.2	EPA RSL	0.1	0.094	0.08	0.0312
Aroclor-1232	11141-16-5	0.17	EPA RSL	0.1	0.066	0.0307	0.0102
Aroclor-1242	53469-21-9	0.23	EPA RSL	0.1	0.066	0.048	0.0182
Aroclor-1248	12672-29-6	0.23	EPA RSL	0.1	0.066	0.015	0.00478
Aroclor-1254	11097-69-1	0.24	EPA RSL	0.1	0.066	0.0333	0.011
Aroclor-1260	11096-82-5	0.24	EPA RSL	0.1	0.066	0.00667	0.00233
Pesticides / 8081							
DDD	72-54-8	1.9	EPA RSL	0.5	0.0034	0.00267	0.00109
DDE	72-55-9	2	EPA RSL	0.5	0.0034	0.00133	0.000476
DDT	50-29-3	1.9	EPA RSL	0.5	0.004	0.00267	0.00118
Aldrin	309-00-2	0.039	EPA RSL	0.015	0.0034	0.00133	0.000502
alpha-BHC	319-84-6	0.086	EPA RSL	0.001	0.0034	0.00133	0.000428
beta-BHC	319-85-7	0.3	EPA RSL	0.1	0.004	0.00267	0.00133
Chlordane (isomers)	57-74-9	1.7	EPA RSL	1	0.0034	0.00133	0.000646
Dieldrin	60-57-1	0.034	EPA RSL	0.01	0.0034	0.00133	0.00042
Endosulfan I [2]	959-98-8	470	EPA RSL	100	0.0034	0.001	0.000352
Endosulfan II [2]	33213-65-9	470	EPA RSL	100	0.0034	0.00133	0.000574
Endrin	72-20-8	19	EPA RSL	9	0.0034	0.00133	0.000612
gamma-BHC	58-89-9	0.57	EPA RSL	0.1	0.0034	0.00133	0.000393
Heptachlor	76-44-8	0.13	EPA RSL	0.1	0.0034	0.00133	0.000418
Heptachlor Epoxide	1024-57-3	0.07	EPA RSL	0.05	0.0034	0.00267	0.000852
Methoxychlor	72-43-5	320	EPA RSL	100	0.0066	0.00267	0.0009
Toxaphene	8001-35-2	0.49	EPA RSL	0.34	0.34	0.0933	0.0316

Notes: [1] EPA RSL (*THQ-= 1.0, Lower value of Carcinogenic SL or Noncarcinogenic SL*) for residential soil, April 2021, applies to all compounds with EPA RSL reference.

[2] See Footnote [1] RSL / Endosulfan, CAS 115-29-7

BHC = hexachloro cyclohexane
CAS = Chemical Abstracts Service
DDD = dichlorodiphenyl dichloroethane
DDE = dichlorodiphenyl dichloroethylene
LOQ = Limit of Quantitation
LOD = Limit of Detection
MDL = Method Detection Limit
mg/kg = milligrams per kilogram

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DDT = dichlorodiphenyl trichloroethane

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Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference[1]	Project Quantitation Limit Goal	Laboratory-Specific Limits		
					LOQ	LOD	MDL
		mg/kg					
Semi volatiles / 8270							
1,2-Dichlorobenzene	95-50-1	1,800	EPA RSL	100	0.33	0.067	0.022
1,4-Dichlorobenzene	106-46-7	2.6	EPA RSL	1	0.33	0.033	0.0136
2,4,5-Trichlorophenol	95-95-4	6,300	EPA RSL	10	0.33	0.033	0.01
2,4,6-Trichlorophenol	88-06-2	49	EPA RSL	10	0.33	0.033	0.01
2,4-Dichlorophenol	120-83-2	190	EPA RSL	10	0.33	0.033	0.01
2,4-Dimethylphenol	105-67-9	1,300	EPA RSL	100	0.33	0.133	0.066
2,4-Dinitrophenol	51-28-5	130	EPA RSL	10	1.6	1	0.333
2,4-Dinitrotoluene	121-14-2	1.7	EPA RSL	1	0.33	0.133	0.066
2,6-Dinitrotoluene	606-20-2	0.36	EPA RSL	0.33	0.33	0.067	0.028
2-Chloronaphthalene	91-58-7	4,800	EPA RSL	10	0.33	0.033	0.01
2-Chlorophenol	95-57-8	390	EPA RSL	10	0.33	0.067	0.021
2-Nitroaniline	88-74-4	630	EPA RSL	10	1.6	0.133	0.05
3,3'-Dichlorobenzidine	91-94-1	1.2	EPA RSL	1	1.6	0.267	0.09
4-Chloroaniline	106-47-8	2.7	EPA RSL	1	0.33	0.267	0.0819
4-Nitroaniline	100-01-6	27	EPA RSL	10	1.6	0.267	0.0725
Benzyl Alcohol	100-51-6	6,300	EPA RSL	100	0.33	0.033	0.01
Bis(2-Chloroethoxy) methane	111-91-1	190	EPA RSL	10	0.33	0.067	0.023
Bis(2-Chloroethyl) Ether[2]	111-44-4	0.23	EPA RSL	0.33	0.33	0.033	0.0166
Bis(2-Ethylhexyl) Phthalate	117-81-7	39	EPA RSL	10	0.33	0.133	0.046
Dibenzofuran	132-64-9	73	EPA RSL	10	0.33	0.067	0.02
Diethyl Phthalate	84-66-2	51,000	EPA RSL	100	0.66	0.067	0.026
Di-N-butyl Phthalate	84-74-2	6,300	EPA RSL	100	0.33	0.067	0.029
Hexachlorobenzene[2]	118-74-1	0.21	EPA RSL	0.33	0.33	0.067	0.029
Hexachlorobutadiene	87-68-3	1.2	EPA RSL	0.33	0.33	0.033	0.01
Hexachloroethane	67-72-1	1.8	EPA RSL	1	0.33	0.067	0.0213
Isophorone	78-59-1	570	EPA RSL	100	0.33	0.067	0.017
Nitrobenzene	98-95-3	5.1	EPA RSL	1	0.33	0.067	0.022
n-Nitrosodi-n-propylamine[2]	621-64-7	0.078	EPA RSL	0.33	0.33	0.167	0.068
Pentachlorophenol[2]	87-86-5	1	EPA RSL	1	1.6	1	0.33
Phenol	108-95-2	19,000	EPA RSL	100	0.33	0.067	0.018

Notes: [1] EPA RSL for residential soil, April 2019, applies to all compounds with EPA RSL reference.
[2] While the LOQ's are above the project comparison criterion, the MDL are below the comparison criteria. The laboratory will report non-detected analytes to the LOD. Detected concentrations b/w the MDL and LOQ as estimated (J).

CAS = Chemical Abstracts Service
LOD = Limit of Detection
LOQ = Limit of Quantitation
MDL = Method Detection Limit
mg/kg = milligrams per kilogram

Analyte	CAS Number	Screening Level	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific Limits		
					LOQ	LOD	MDL
		mg/kg					
Polycyclic Aromatic Hydrocarbons (PAH's) / 8270-SIM							
Acenaphthene	83-32-9	3,600	EPA RSL1	10	0.01	0.00107	0.00032
Anthracene	120-12-7	18,000	EPA RSL	10	0.01	0.00433	0.00144
Benz(a)anthracene	56-55-3	1.1	EPA RSL	1	0.01	0.00433	0.0018
Benzo(a)pyrene	50-32-8	0.11	EPA RSL	0.1	0.01	0.00433	0.00148
Benzo(b)fluoranthene	205-99-2	1.1	EPA RSL	0.1	0.01	0.00667	0.0024
Benzo(k)fluoranthene	207-08-9	11	EPA RSL	0.1	0.01	0.00433	0.002
Chrysene	218-01-9	110	EPA RSL	10	0.01	0.00433	0.002
Dibenz(a,h)anthracene	53-70-3	0.11	EPA RSL	0.1	0.01	0.00667	0.0026
Fluoranthene	206-44-0	2,400	EPA RSL	100	0.01	0.00433	0.002
Fluorene	86-73-7	2,400	EPA RSL	100	0.01	0.00267	0.00094
Indeno(1,2,3-cd) pyrene	193-39-5	1.1	EPA RSL	0.1	0.01	0.00667	0.0022
Naphthalene	91-20-3	3.8	EPA RSL	1	0.01	0.002	0.000652
Pyrene	129-00-0	1,800	EPA RSL	10	0.01	0.00667	0.0022

Notes: [1] EPA RSL for residential soil, April 2019, applies to all compounds with EPA RSL reference.

CAS = Chemical Abstracts Service
LOQ = Limit of Quantitation
LOD = Limit of Detection
MDL = Method Detection Limit
mg/kg = milligrams per kilogram

Analyte	CAS Number	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific Limit		
					LOQ	LOD	MDL
		mg/kg					
Total Petroleum Hydrocarbons (TPH) / 8015							
TPH-gasoline range organics (GRO)	NA	3,500 Total TPH (soil and sediment)	HPNS TPH Action Limit	100	2	2	0.759
TPH-diesel range organics (DRO)	NA			100	8	8	3.64
TPH-motor oil range organics (MRO)	NA			200	24	23.4	7.82

Notes: CAS = Chemical Abstracts Service
HPNS = Hunters Point Naval Shipyard
LOQ = Limit of Quantitation
LOD = Limit of Detection
MDL = Method Detection Limit
mg/kg = milligrams per kilogram

Analyte	CAS Number	Units	Project Comparison Criteria	Project Comparison Criteria Reference	Project Quantitation Limit Goal	Laboratory-Specific	
						QL	MDL
Asbestos / CARB 425							
Asbestos	-	Percent	< 0.25	CARB	0.25	0.25	Not Applicable

Notes: CAS = Chemical Abstracts Service
MDL = Method Detection Limit
QL = quantitation limit

SAP WS #16: PROJECT SCHEDULE / TIMELINE TABLE

Activities	Organization	Dates		Deliverable
		Estimated Date of Initiation	Estimated Date of Completion	
Internal Draft SAP Preparation	EIP	October 2019	April 2020	Internal Draft SAP
Navy BRAC/RASO SAP Review	Navy BRAC and RASO	May 2021	May 2021	Comments and responses
Navy QAO SAP Review	Navy QAO	May 2021	June 2021	Comments and responses, Navy chemist signature
Regulatory Review	EPA, DTSC, CDPH, City of San Francisco	June 2021 / March 2022	August 2021 / May 2022	Comments and responses,
Draft-Final SAP	EIP	August 2021 / May 2022	September 2021 / June 2022	Draft Final SAP
Final SAP	Navy and regulatory agencies	September 2021 / June 2022	October 2021 / July 2022	Final SAP
Subcontracting/Procurement	EIP	December 2021 / November 2022	January 2022 / December 2022	Subcontractor contracts
Field Investigations	EIP	February 2022 / November 2022	November 2022	None
Laboratory Analyses; Data Validation and Verification; and Data Management	Eurofins Test America – St Louis; Laboratory Data Consultants; EIP	February 2022	November 2022	Analytical and data validation reports
Internal Draft Report	EIP	December 2022	January 2023	Draft reports
Navy BRAC/RASO Report Review	Navy BRAC and RASO	January 2023	February 2023	Comments and responses
Draft Report	EIP	February 2023	March 2023	Draft Report
Regulatory Report Review	EPA, DTSC, CDPH, City of San Francisco	March 2023	July 2023	Comments and responses
Draft-Final report	EIP	July 2023	August 2023	Draft-Final Report
Regulatory Report Review	EPA, DTSC, CDPH, City of San Francisco	August 2023	September 2023	Comments and Responses
Final Report	Navy and regulatory agencies	September 2023	October 2023	Final report

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SAP WS #17: SAMPLING DESIGN AND RATIONALE

The Parcel E evaluation survey, sampling, and analytical program, as well as the rationale for selecting sample locations, is described as below.

17.1 SOIL CONFIRMATION INVESTIGATION

This section describes the design of radiological confirmation investigations, including gamma scanning and soil sample collection in soil. The radiological confirmation investigation design and rationale are primarily based on methods, techniques, and instrument systems in the Basewide Radiological Management Plan (TtEC, 2012), with the ultimate requirement to demonstrate compliance with the Parcel E ROD RAO (Navy, 2009).

A two-phased approach is planned for the soil confirmation investigation for the surface and subsurface TU soil associated with former sanitary and storm drain lines. The approach is based on a proposal by the regulatory agencies to achieve a high level of confidence that the Parcel E ROD RAO has been met for soil. For Phase 1, 100 percent of soil will be re-excavated and characterized for the Parcel TUs identified in **Table 17-2**. Soil sampling and scanning at the remaining TUs will be performed as part of Phase 2 to increase confidence that current site conditions comply with the Parcel E ROD RAO. Evaluation of the results of Phase 1 may lead to Phase 2 TUs if contamination is identified in Phase 1 trenches.

For surface soil areas associated with soil from building sites, radiological confirmation investigation will be conducted at the designated SU's in Parcel E. The name, size, and boundary of the TUs and SUs will be based on the previous plans and reports. EIP will utilize the previous established SU location grids designations, when possible.

Regardless of the survey technique used, the samples will be shipped to the off-site laboratory for gamma spectroscopy analysis and 10 percent for GFPC analysis for ^{90}Sr. If the results indicate that the ^{226}Ra, ^{137}Cs and ^{90}Sr concentrations in the soil are below RGs, the soil will be returned to TU of origin.

If ^{90}Sr or ^{137}Cs is detected above release criteria, then additional analysis for isotopic ^{238}Pu/^{239}Pu/^{240}Pu or other alpha emitters may be performed to confirm contamination. If ^{226}Ra gamma spectroscopy concentrations exceed the RG and the range of expected NORM concentrations, then the soil sample will be analyzed for ^{234}U, ^{238}U, and ^{226}Ra using alpha spectroscopy. If the concentrations of the radionuclides in the uranium natural decay series are consistent with the assumption of secular equilibrium, then the ^{226}Ra concentration is NORM and site conditions comply with the ROD RAO.

ESU and SFU soil that has confirmed clean will be available to use as fill material in the TU from which it was excavated from.

The ROCs for the soil areas are listed in **Table 17-1** and **Table 17-4**, and RGs are listed in WSs #15.1, #15.2, #15.3, and #15.4. Samples collected in support of the TU, SU, and Building Survey soil confirmation investigations are provided in this worksheet. The analyses will be based on the site-specific ROCs listed on **Table 17-1** and **Table 17-4**.

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Table 17.1: Radionuclides of Concern

		Primary Radionuclides of Concern							
		Cesium 137	Radium 226	Strontium 90	Plutonium 239	Uranium 235	Americium 241	Cobalt 60	Tritium
Soil Area		^{137}Cs	^{226}Ra	^{90}Sr	^{239}Pu	^{235}U	^{241}Am	^{60}Co	^{3}H
Former Sanitary Sewer and Storm Drain Lines -	*Trench Unit (TU)*	x	x						
Former Building Sites -	*Survey Unit (SU)*	x	x	x	x	x	x	x	x
Building Surveys		x	x	x	x				

Secondary ROC analyses include:

- Uranium-234 [^{234}U], Uranium-238 (^{238}U],
- Plutonium-238 [^{238}P] and Plutonium-240 [^{240}P]

Refer to **Figures 3, 4**, and **5** for additional Radionuclide analyses that may be required.

17.1.1 Phase 1 Trench Unit

The Phase 1 soil confirmation investigation from each TU will include:

- A gamma scan of 100 percent of soil.
- Collection of the required systematic and biased soil samples (based on gamma scan measurements).

	Radionuclides of Concern	
	Cesium 137	Radium 226
Soil Area	^{137}Cs	^{226}Ra
Former Sanitary Sewer and Storm Drain Lines	x	x

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- The excavation limits of each Phase 1 TU will initially be excavated to the original excavation limits, which will be evaluated in approximately 298 cubic yards of excavated soil units (ESU's).
- A (approximated) 6-inch over excavation, designated as the sidewall floor unit (SFU), outside of the previously established ESU boundaries. The over excavation will include the sidewalls and trench floor. The SFU exhumed material will be maintained and tracked separately from the ESUs.

The gamma-scanned of the excavated soil will be performed by one of two methods, Radiological Screening Yard (RSY) or Soil Sorting System technology (S3). EIP has selected to the use the RSY pad option which utilizes a surface scan. Each ESU/SFU, once transported to the designated RSY pad, will be spread out to a maximum depth 9 inches.

17.1.1.1 Gamma Scan Surveys

The excavated TU material will be assayed using the RSY pad process. Each ESU/SFU, once transported to the designated RSY pad, will be spread out to a maximum depth 9 inches in preparation for the gamma scan survey. 100% of RSY pad surfaces will be surveyed using the Eagle iScan (or equivalent) towable scanning process.

If gamma scan surveys indicate areas of potentially elevated activity in soil above the investigation levels, an investigation of the potential area of elevated activity will be initiated. At a minimum, the contractor will further evaluate the gamma scan data and collect biased soil samples. Further details on this process are described in the work plan.

17.1.1.2 Soil Sampling

A minimum of 14 systematic samples will be collected from each ESU and SFU. At a minimum a single biased sample will be collected from the location that had the highest gamma scan reading, additional biased soil samples may be collected from locations if readings exceed the applicable RG. The soil samples will be analyzed for the applicable ROC analysis (**Table 17-2**) by an accredited offsite laboratory.

The systematic soil samples will be located using Visual Sample Plan (VSP) software (or equivalent). Each RSY (ESU or SFU) will be mapped into VSP, such that at a minimum, 14 systematic soil samples will be collected in the SU. The systematic soil samples will be plotted using a random start triangular grid using the VSP software with GPS coordinates for each systematic sample. The RSY soil confirmation samples will be collected following the procedure described in the SAP WS#21.

- ESU: The systematic soil samples will be plotted using a random start triangular or square grid using the visual sample plan software.
- SFU: Each 1,000-cubic-meter trench SFU will be plotted using visual sample plan software (or equivalent) to determine the location of the 14 systematic soil samples.

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Biased soil sample(s) will be collected from the approximate location(s) of the highest elevated gamma scan survey measurement (required in both the ESU and SFU). If the scan does not reveal a reading above the RG's, then a single biased sample will be collected at the location where the highest reading was recorded. If areas displaying elevated activity are collocated, an attempt will be made to locate the area with the highest gamma scan results and designate it as the biased sample location to represent the collocated elevated areas.

If the investigation results collected during the gamma scan surveys, systematic soil samples, and the biased soil samples of the over-excavated material (SFU) demonstrate exceedances of the RGs (and are not attributed to NORM or anthropogenic background) the material will be segregated for further evaluation prior to backfill. The systematic sample evaluation/comparison of the results will be performed against the RG and background, and whichever is higher.

If required, the SFU investigation will include the performance of a gamma scan over the trench surface requiring investigation and possible additional biased and systematic sampling. The gamma scan will be performed in two stages. The first stage is a 100 percent gamma scan of the accessible areas. Review of the *in situ* gamma scan data will determine whether further investigation is warranted. If further investigation is not warranted, then the second stage is not necessary, and systematic samples will be collected. If further investigation is warranted, then biased samples may be collected. A minimum of 14 systematic soil samples will be collected from each *in situ* SFU requiring further investigation. The *in situ* soil samples will be collected from the trench surface at a depth of 0 to 6 inches.

17.1.2 Phase 2 Trench Confirmation Investigation

Investigations of the Phase 2 trenches will consist of a combination of gamma scan surveys and soil samples. Phase 2 of the investigation evaluates the remaining TUs (**Table 17-3**). Soil samples shall be collected with a drilling or direct-push rig. One soil boring will be located every 50 linear feet along the former trench sidewalls. If further action (i.e., excavation of the TUs) is required for the Phase 2 TUs, then they will follow the same procedures as identified and performed on the Phase 1 TUs.

17.1.2.1 Gamma Scan Surveys

Gamma scan surveys of the surface soil will be performed using the same protocols and methods as those in used at the RSY pads. Of the accessible surface of the Phase 2 trenches, 100 percent will be gamma scan-surveyed.

For the Phase 2 TUs, if gamma scan surveys indicate areas of potentially elevated activity in soil above the soil confirmation investigation levels, then an investigation of the potential area of elevated activity will be initiated. At a minimum, the contractor will further evaluate the gamma scan data and collect biased soil samples. The biased soil sample will be collected from the approximate location of the highest elevated gamma scan survey measurement. If areas displaying elevated activity are collocated, then an attempt will be made to locate the area with the highest gamma scan results and designate it as the biased sample location to represent the collocated elevated areas.

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Table 17-2: Phase 1 Soil Trench Units

Former Trench Unit Name	Excavation of Original Trench Unit		Sidewalls + Floor		Total		
	Estimated Volume of Original Excavation (cubic yards)[a]	Number of Excavation Soil Units[b]	Estimated Volume of 6-Inch Over-Excavation of Sidewalls and Floor (cubic yards)	Number of Sidewall Floor Units[b]	Volume (cubic yards)	Number of Units	Estimated Number of Systematic Samples[c]
TU-152	494	2	135	1	629	3	42
TU-158	612	3	161	1	773	4	56
TU-159	364	2	114	1	478	3	42
TU-162	442	2	124	1	566	3	42
TU-163	636	3	166	1	802	4	56
TU-214	775	3	191	1	966	4	56
TU-217	1,080	4	193	1	1,273	5	70
TU-222	450	2	176	1	626	3	42
TU-223	795	3	184	1	979	4	56
TU-235	411	2	154	1	565	3	42
TU-245	942	4	133	1	1,075	5	70
TU-246	1,152	4	139	1	1,291	5	70
TU-300	1,056	4	187	1	1,243	5	70
TU-301	1,220	5	193	1	1,413	6	84
TU-307	1,080	4	190	1	1,270	5	70
TU-340	906	4	179	1	1,085	5	70
TU-341	1,604	6	162	1	1,766	7	98
TU-342	1,216	5	177	1	1,393	6	84
TU-345	1,597	6	187	1	1,784	7	98
TU-346	1,157	4	191	1	1,348	5	70

Total Excavation Volume (cubic yards): 21,325

Total Number of Units: 92

Total Number of Systematic Samples: 1,288

Notes: [a] The estimated volume of the original excavation was derived using the Estimated Backfill Quantity information provided in Table 1 of the Performance Work Statement, Radiological Confirmation Sampling and Survey at Parcel E, Former Hunters Point Naval Shipyard, San Francisco, California.

[b] The number of Excavation Soil Units and Sidewall Floor Units are calculated from dividing the estimated volume of the excavation by 298 yd^3, which is based on a volume of 1,000 m^2 x 9 inches = 298 yd^3 (~327 tons of soil).

[c] Assumes 14 systematic samples in each survey unit.

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Table 17-3: Phase 2 Trenches

Trench Unit	Surface Area (square feet)[a]	Number of Systematic Borings in Original TU Material	Number of Samples in Original TU Material	Number of Borings from Sidewalls and Bottom[b]	Number of Samples from Sidewalls and Bottom
TU-154	323	14	42	2	6
TU-155	291	14	42	2	6
TU-156	5,134	14	42	12	36
TU-157	9,020	14	42	24	72
TU-160	8,267	14	42	18	54
TU-161	7,319	14	42	22	66
TU-165	710	14	42	2	6
TU-201	3,132	14	42	8	24
TU-215	10,290	14	42	28	84
TU-216	10,290	14	42	16	48
TU-218	9,139	14	42	18	54
TU-224	9,827	14	42	24	72
TU-225	10,409	14	42	2	6
TU-228	10,247	14	42	22	66
TU-229	10,183	14	42	10	30
TU-230	9,623	14	42	12	36
TU-240	1,238	14	42	4	12
TU-241	6,114	14	42	18	54
TU-248	9,914	14	42	18	54
TU-249	10,183	14	42	22	66
TU-305	10,430	14	42	36	108
TU-306	9,838	14	42	26	78
TU-308	10,312	14	42	20	60
TU-309	10,226	14	42	22	66
TU-310	8,686	14	42	18	54
TU-311	3,950	14	42	6	18
TU-343	9,558	14	42	18	54
TU-344	9,160	14	42	16	48
TU-347	10,247	14	42	20	60
TU-348	10,506	14	42	12	36
TU-349	10,064	14	42	10	30
TU-350	7,083	14	42	10	30
TU-351	6,243	14	42	10	30
TU-352	7,567	14	42	12	36
TU-353	4,585	14	42	10	30
TU-354	5,554	14	42	14	42
TU-355	4,133	14	42	12	36
	Sample Subtotals:		**1,554**		**1,668**

Total Number of Systematic Samples (Original TU Material + Sidewalls and Bottom) is 3,222

Notes [a]From Table 2 of the Performance Work Statement, Radiological Confirmation Sampling and Survey at Parcel E, Former Hunters Point Naval Shipyard, San Francisco, California.
[b]Assumes a boring every 50 linear feet of trench on each sidewall

17.1.2.2 Systematic Borings Process

The systematic boring locations will be cored down to approximately 6 inches below the depth of the previous excavation within each trench boundary. Soil samples will be collected as described in WS#14, WS#23, and this section. The SS and SD lines were sometimes installed on bedrock, in these situations, sampling of bedrock will not be performed. If refusal is encountered within 6 inches of the expected depth of the trench, then the soil sample will be collected from the deepest section of the core. If refusal is encountered more than 6 inches above the expected depth of the trench, then the sample location will be moved to avoid the subsurface obstruction. Additionally, systematic samples will be collected from sidewall locations every 50 linear feet on alternating sides, representative of each of the trench sidewalls. The boring locations will be located within 1 meter of the previous sidewall excavation limits and will extend to the maximum previous excavation depth. In the same action described in the previous paragraph, core sections will be retrieved, scanned, and sampled such that at least three samples will be collected from each of the six sidewall boring locations. The GPS data for each soil boring will be recorded.

To acquire three samples from each boring, one surface and one floor sample will be collected from each sample core. The sample cores will be scanned for gamma radiation along the entire length of each core using a Ludlum Model 44-20 3-inch by 3-inch sodium iodide (or equivalent). Scan measurement results will be evaluated against the investigation level to identify core sections with elevated gamma radiation. The investigation level for a gamma instrument is the RBA mean plus three standard deviations. Once the RBA measurements are completed by the instrumentation, EIP will be able to provide regulatory agencies our instrument-specific ILs. Core sections that exceed the investigation level will have biased soil samples collected to investigate the potential for small areas of elevated activity in fill. If no core section exceeds the investigation level, then a biased sample will be collected from the core segment with the highest gamma scan reading that was not already sampled, for a total of at least three samples from each core. If further action is required for the Phase 2 TUs, then the same procedures as identified and performed on the Phase 1 TUs will be followed.

17.1.3 Former Building Sites

Investigations of the former building sites consist of a combination of gamma scan surveys and the collection of soil samples. The gamma scan surveys will be performed within the footprint of the buildings and surface soil samples will be collected from each site. The former building sites included in this study are: 517, 503, 520, 506, 508, 510/510A, 509, 529, IR-04 Former Scrap Yard/Former Building 807 Site, 704, 704 Storage Yard, and Former Shacks 79 and 80.

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17.1.3.1 Gamma Scan Surveys

Gamma scan surveys of the surface soil will be performed using the same protocols and methods as those in the RSY pads. Of the accessible surface of the Survey Units, 100 percent will be gamma scan surveyed using a global positioning system coupled to a large volume gamma scintillator, equipped with real time gamma spectroscopy and data logging.

If gamma scan surveys indicate areas of potentially elevated activity in soil above the investigation levels, then an investigation of the potential area of elevated activity will be initiated. At a minimum, the contractor will further evaluate the gamma scan data and collect biased soil samples. The biased soil sample will be collected from the approximate location of the highest elevated gamma scan survey measurement. If areas displaying elevated activity are collocated, then an attempt will be made to locate the area with the highest gamma scan results and designate it as the biased sample location to represent the collocated elevated areas.

17.1.3.2 Radiologic Soil Samples

The former building site soil samples will be collected at the rate of 14 samples per survey unit. Additionally, a minimum of one biased soil sample will be collected from the SU pad where discrete areas of elevated gamma count rate readings that exceed instrument specific investigation levels that are not attributable to naturally occurring radioactivity or where sample results exceeding the project screening criteria for ROCs are observed.

The systematic soil sample locations will be positioned throughout the Screening unit by first randomly selecting a starting point and establishing a systematic pattern. The triangular grid is also utilized for the SU pads because it is generally more efficient for locating small areas of elevated activity. Grid coordinates are laid out using the hand-held global positioning system (or equivalent). The soil confirmation samples will be collected following the procedure described in the SAP WS#21.

The systematic soil samples will be collected in the same format as the RSY Phase 1and Phase 2 TUs. The systematic soil samples will be located using Visual Sample Plan (VSP) software (or equivalent) once the survey boundaries have been established. Each SU will be mapped in VSP, such that at a minimum, 14 systematic soil samples will be collected in the SU. The systematic soil samples will be plotted using a random start triangular grid using the VSP software with GPS coordinates for each systematic sample. The sample location map, denoting the location of each sample with the SU, will be generated following the completion of the field activities.

Table 17-4 identifies the ROCs for each former building site and **Table 17-5** list the building IDs and their associated survey units. The locations of the former building sites are included in **Figure 2**.

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Table 17-4: Radionuclides of Concern (listed by Building)

Building	ROCs	Reference
Former Building Site 517	Cs-137, Sr-90, Co-60 and Ra-226	
Former Building Site 503	Cs-137, Sr-90 and Ra-226	
Former Building Site 520	Cs-137, Sr-90 and Ra-226	
Former Building Site 506	Cs-137, Sr-90, Pu-239, Am-241, and Ra-226, H-3	
Former Building Site 508	Cs-137, Sr-90 and Ra-226	
Former Building Site 510	Cs-137, Sr-90, Pu-239 and Ra-226	
Former Building Site 529	Cs-137, Sr-90, H-3 and Ra-226	
IR-04 Former Scrap Yard and Former Building 807 Site	Cs-137, Sr-90 and Ra-226	
Former Building Site 704	Cs-137, Sr-90, Pu-239 and Ra-226	
Former Building Site 704 Storage Yard	Cs-137, Sr-90, Pu-239 and Ra-226	
Former Building Site 707 Triangle	Cs-137, Sr-90, Pu-239, U-235 and Ra-226	
Former Building Site 500 Series	Cs-137, Sr-90, Pu-239, Am-241 and Ra-226	
Former Building Site Shack 79 and 80	Cs-137, Sr-90 and Ra-226	

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Table 17-5: Systematic Samples per Former Building Site

Former Building Site	Former Class 1 Survey Unit Name	Former Class 2 Survey Unit Name	Area (square meters)	Number of Systematic Samples
Former Building Site 517	SU1 and SU2	-	2,249	28
Former Building Site 503	SU1 – SU24	-	32,669	336
Former Building Site 520	SU1 – SU5	-	3,246	70
Former Building Site 506	SU1 – SU5	-	3,640	70
Former Building Site 508	SU1	-	1,496	14
Former Building Site 510	SU1	-	1,914	14
Former Building Site 529	SU1	-	392	14
IR-04 Former Scrap Yard and Former Building 807 Site	SU1 – SU9	SU10	7,040	140
Former Building Site 704	SU1 and SU3	SU4	1,219	42
Former Building Site 704 Storage Yard	SU1, SU3, and SU4	SU7	1,384	70
Former Building Site 707 Triangle	SU1 – SU23	-	19,967	322
Former Building Site 500 Series	SU1 – SU27	-	41,473	378
Former Building Site Shack 79 and 80	SU1, SU2, and SU3	-	2,591	42
		Total Number of Systematic Samples		**1,540**

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17.1.4 Existing Building Surveys

The confirmation investigations of the existing building sites consist of position-sensitive proportional counters (PSPCs), gas proportional detectors/counters (GPD), scintillation detectors, and the collection of swipe samples. The surveys will be conducted in buildings 406, 414, 521, and 810 and the locations of the building can be found in **Figure 2**. Refer to **Table 17-6** for the ROCS applicable to these buildings. EIP will utilize the previous established SU location grids designations, when possible, along with the Class 1, Class 2, and Class 3 SU location descriptions.

Fourteen (14) systemic and potentially biased static measurements will be performed per SU. Prior to the static measurement and swipe sample collection, the location of the sample will be recorded. The swipe samples will be taken at all locations that the systematic and biased static measurements were collected. They will be taken dry, using moderate pressure, over an area of approximately 100 cm^2. The wipe samples collected will not be submitted for off-site analysis. All analyses will be performed on-site by project personnel and the readings will be recorded. **Table 17-7** identifies the equipment that will be used and **Table 17-8** identifies the sample collection details for each building. The survey unit scan and swipe sample locations, designated via the CSP software, will be based on the building survey data and building dimensions provided in the work plan.

Table 17-6: Existing Building Surveys - Radionuclides of Concern

	Radionuclide of Concern			
	Cesium 137	Radium 226	Strontium 90	Plutonium 239
Existing Building Number	^{137}Cs	^{226}Ra	^{90}Sr	^{239}Pu
406	x	x		
414		x		
521	x	x	x	x
810	x	x	x	

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Table 17-7: Survey Scan Information

Survey/Scan Type		Radiation Type	Instrument Type(s): Manufacturer (Model Number)	Size of Scan	Notes
Position-sensitive Proportional Counters (PSPC)	Surface and Static Measurements	alpha and beta	Radiation Safety and Control Services, Inc. Surface Scan Monitor (SCM)	Total area per scan 100 cm^2 (via 4 x 25 cm^2 scans/rolls)	N/A
Gas Proportional Detectors (GPD)	Surface	-	Ludlum (Model 43-47)	584 cm^2 (2.5 x 15.9 x 46.4 cm)	Data logged via Ludlum Model 2360 or equivalent
Gas Proportional Detectors (GPD)	Surface and Static Measurements	alpha and beta	Ludlum (Model 43-68)	126 cm^2 (10 x 11.7 x 19.8 cm)	N/A
Scintillation Detectors (SD)	Scanning and Static Measurements	alpha and beta	Ludlum (Model 43-96)	100 cm^2	N/A
Swipe Sample (SW)	Surface	alpha and beta	Ludlum (Model 3030) or equivalent	20.3 cm^2	Utilizes 5.1 cm swipe papers loaded into singe sample tray

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Table 17-8: Existing Building Survey Unit Information

Existing Building Number	Survey Units (SUs)									Limiting Alpha Emitting ROC	Limiting Beta Emitting ROC	Estimated Number of Static Measurements [1]	Estimated Number of Swipes[3]	Associated Work Plan Figure
	Class 1			Class 2			Class 3							
	Number of SUs	SU IDs	Locations	Number of SUs	SU IDs	Locations	Number of SUs	SU IDs	Locations					
406	42	SU-01 thru SU-42	concrete perimeter lower walls and asphalt floors	1	SU-43	all the concrete perimeter upper walls and the ceiling.	0	-	-	Not ID'd	Not ID'd	774	774	4-2
414	19	SU-01 thru SU-19	concrete flooring, concrete support columns, and concrete perimeter lower walls	1	SU-20	all the concrete perimeter upper walls	0	-	-	^{226}Ra	^{90}Sr	360	360	4-3
521	6	SU-01 thru SU-06	concrete flooring and sheet metal (perimeter) or sheetrock (interior) lower walls	1	SU-07	-	1	SU-08	-	^{226}Ra	^{90}Sr	144	144	4-4
810	28	SU-01 thru SU-28	concrete flooring, wooden or concrete perimeter lower walls	1	SU-29	wooden or metal flooring and wooden perimeter lower walls	0	-	-	^{226}Ra	^{90}Sr	522	522	4-5
BG [2]	14 readings per identified material: Asphalt, Concrete, Metal, Wood, Sheet Rock											90	90	-
											Total	1,890	1,890	

[1] 14 readings per SU
[2] Background: The number of readings will increase if additional material, not identified above, is encountered (Section 4.6.3.1 of WP)
[3] Swipe samples will be taken at the locations of systematic and biased static measurements (Section 4.6.5 of WP)

17.2 Imported Fill

If imported fill material is required, the borrow source will be sampled and submitted for approval, prior to use as backfill material at Parcel E. Per the DTSC "Information Advisory on Clean Import Fill Material" (2001) the following sampling frequency/requirements will be utilized:

Area of Individual Borrow Area	Sampling Requirements
Up to 2 acres	Minimum of four samples
2 to 4 acres	Minimum of one sample every half acre
4 to 10 acres	Minimum of eight samples
Greater than 10 acres	Minimum of eight locations with four subsamples

Volume of Borrow Area Stockpile	Samples per Volume
Up to 1,000 cubic yards	One sample per 250 cubic yards
1,000 to 5,000 cubic yards	Four samples for first 1,000 cubic yards plus one sample per each additional 500 cubic yards
Greater than 5,000 cubic yards	12 samples for first 5,000 cubic yards plus one sample per each additional 1,000 cubic yards

Import fill soil samples will be analyzed for the following parameters:

- VOCs — EPA Methods 8260B
- SVOCs — EPA Method 8270C
- PAHs — EPA Method 8270C-SIM Pesticides — EPA Method 8081A
- PCBs — EPA Method 8082A
- TPH (GRO, DRO, MRO) — EPA Method 8015B
- California Code of Regulations Title 22 Metals (17) — EPA Methods 6020/7471A
- Asbestos — CARB 435 (one per source)
- Gamma Spectroscopy by EPA Method 901.1
- Total Sr/^{90}Sr, GFPC — EPA Method 905.0 or equivalent

Analytical results from the import material will be compared to the criteria listed in WS #15.

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Only soil or fine materials will be sampled. If imported engineered fill (i.e., crushed rock and aggregate base) is used, then the engineered fill will not be sampled for chemical analyses, but fines will be screened for asbestos by CARB Method 435, and total metals (EPA Method 6020/7471A), in accordance with recommendations of the *Information Advisory on Clean Import Fill Material* (DTSC, 2001). Engineered fill will also be sampled for ^{226}Ra to verify that it does not exceed Hunters Point Naval Shipyard background limits. If the quarry or source of rock cannot supply the required asbestos or metals screening information, then one sample of gravel or rock fines will be collected from each fill source. If the results are below the criteria, then the material may be used for import. Otherwise, another source will be identified and sampled.

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SAP WS #18: SAMPLING LOCATIONS AND METHODS / SOP REQUIREMENTS TABLES

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SAP WS #18.1: Sampling Locations and Methods / SOP Requirements Table – Phase 1

Sampling Location	Type	Sample ID Number[1]	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Sampling SOP Reference
Phase 1 Trench Unit							
TU-152	Excavated Soil Unit (ESU)/ Side Wall or Floor Unit (SFU)	HPPE-ESU-152X-XXX / HPPE-SFU-152X-XXX	Soil	Excavated Material represent in the sidewalls and bottoms of TU (depth varies depending on historical excavated depth).	Gamma Spec GFPC (90Sr) Alpha Spec (as needed)	See Table 17-2	See WS#21
TU-158		HPPE-ESU-158-XXX / HPPE-SFU-158-XXX	Soil				
TU-159		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-162		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-163		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-214		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-217		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-222		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-223		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-235		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-245		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-246		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil			See Table 17-1C	See WS#21

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Sampling Location	Type	Sample ID Number[1]	Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Sampling SOP Reference
TU-300	Excavated Soil / Side Wall or Floor Sample	HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil			See Table 17-1C	See WS#21
TU-301		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-307		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-340		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				
TU-341		HPPE ESU 158 XXX / HPPE-SFU-158-XXX	Soil				

[1] Refer to WS #27 for further details on sample ID nomenclature.

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SAP WS #18.2: SAMPLING LOCATIONS AND METHODS / SOP REQUIREMENTS TABLE - Phase 2

TU Name	Type	Sample ID Number	Matrix	Depth (feet bgs)	Maximum Trench Depth [1]	Analytical Group	Number of Samples	Sampling SOP Reference
TU-154	Soil Boring	HPPE-154-T-SB##-DDDD	Soil	Three samples per boring: • Surface (0 to 6 inches) • 6 inches below the previous excavation depth • One biased sample within the core	4	Gamma Spec GFPC (^{90}Sr) Alpha Spec (as needed)	See Table 17-2	See WS#21
TU-155	Soil Boring	HPPE-155-T-SB##-DDDD	Soil		5			
TU-156	Soil Boring	HPPE-156-T-SB##-DDDD	Soil		7			
TU-157	Soil Boring	HPPE-157-T-SB##-DDDD	Soil		7			
TU-160	Soil Boring	HPPE-160-T-SB##-DDDD	Soil		8			
TU-161	Soil Boring	HPPE-161-T-SB##-DDDD	Soil		6			
TU-165	Soil Boring	HPPE-165-T-SB##-DDDD	Soil		7			
TU-201	Soil Boring	HPPE-201-T-SB##-DDDD	Soil		5			
TU-215	Soil Boring	HPPE-215-T-SB##-DDDD	Soil		8			
TU-216	Soil Boring	HPPE-216-T-SB##-DDDD	Soil		9			
TU-218	Soil Boring	HPPE-218-T-SB##-DDDD	Soil		8			
TU-224	Soil Boring	HPPE-224-T-SB##-DDDD	Soil		8			
TU-225	Soil Boring	HPPE 225-T-SB##-DDDD	Soil		8			
TU-228	Soil Boring	HPPE 228-T-SB##-DDDD	Soil		8			
TU-229	Soil Boring	HPPE 229-T-SB##-DDDD	Soil		10			
TU-230	Soil Boring	HPPE 230-T-SB##-DDDD	Soil		9			
TU-240	Soil Boring	HPPE-240-T-SB##-DDDD	Soil		7			
TU-241	Soil Boring	HPPE-241-T-SB##-DDDD	Soil		7			
TU-248	Soil Boring	HPPE-248-T-SB##-DDDD	Soil		8			
TU-249	Soil Boring	HPPE-249-T-SB##-DDDD	Soil		9			
TU-305	Soil Boring	HPPE-305-T-SB##-DDDD	Soil		7			
TU-306	Soil Boring	HPPE-306-T-SB##-DDDD	Soil		7			
TU-308	Soil Boring	HPPE-308-T-SB##-DDDD	Soil		8			

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TU Name	Type	Sample ID Number	Matrix	Depth (feet bgs)	Maximum Trench Depth [1]	Analytical Group	Number of Samples	Sampling SOP Reference
TU-309	Soil Boring	HPPE-309-T-SB##-DDDD	Soil		7		See Table 17-2	See WS#21
TU-310	Soil Boring	HPPE-310-T-SB##-DDDD	Soil		8			
TU-311	Soil Boring	HPPE-311-T-SB##-DDDD	Soil		10			
TU-343	Soil Boring	HPPE-343-T-SB##-DDDD	Soil		10			
TU-344	Soil Boring	HPPE-344-T-SB##-DDDD	Soil		9			
TU-347	Soil Boring	HPPE-347-T-SB##-DDDD	Soil		9			
TU-348	Soil Boring	HPPE-348-T-SB##-DDDD	Soil		9			
TU-349	Soil Boring	HPPE-349-T-SB##-DDDD	Soil		9			
TU-350	Soil Boring	HPPE-350-T-SB##-DDDD	Soil		9			
TU-351	Soil Boring	HPPE-351-T-SB##-DDDD	Soil		8			
TU-352	Soil Boring	HPPE-352-T-SB##-DDDD	Soil		9			
TU-353	Soil Boring	HPPE-353-T-SB##-DDDD	Soil		7			
TU-354	Soil Boring	HPPE-354-T-SB##-DDDD	Soil		7			
TU-355	Soil Boring	HPPE-355-T-SB##-DDDD	Soil		6			

T = Type

- C= center of trench (one of 14 systematic samples)
- S = sidewall boring sample –(collected every 50', will vary per trench)

= soil boring number - numerical values (01, 02, etc)
HPPE = Hunters Point Parcel E
DDDD = depth of sample (listed in feet)

Example: Surface – 0.5' = 0005
0.5' - 1.0' = 0510
2.5' - 3.0' = 2530

BGS = below ground surface

1) The maximum trench depth was transcribed from the Table 2 of the PWS.
2) Soil samples will be collected per details discussed on WS #17.

SAP WS #18.3: Sampling Locations and Methods / SOP Requirements Table - Former Building Surveys

Former Building Site	Survey Unit Number (Range) *[NN]*	Sample ID Number	Analytical Group	Estimated Number of Systemic Samples	Sampling SOP Reference
517	01 and 02	HPPE-517-SUNN-DDD	See WS#17	28	WS#21
503	01 through 24	HPPE-503-SUNN-DDD	See WS#17	336	
520	01 through 05	HPPE-520-SUNN-DDD	See WS#17	70	
506	01 through 05	HPPE-506-SUNN-DDD	See WS#17	70	
508	01	HPPE-508-SUNN-DDD	See WS#17	14	
510	01	HPPE-510-SUNN-DDD	See WS#17	14	
529	01	HPPE-529-SUNN-DDD	See WS#17	14	
IR-04 Former Scrap Yard and Former Bldg 807 Site	01 thru 10	HPPE-IR04-SUNN-DDD / HPPE-807-SUNN-DDD	See WS#17	140	
704	01 through 03	HPPE-704-SUNN-DDD	See WS#17	42	
704 (Storage Yard)	01, 03, 04, 06, 07	HPPE-704SY-SUNN-DDD	See WS#17	70	
707 Triangle	01 through 23	HPPE-707T-SUNN-DDD	See WS#17	322	
500 Series	01 through 27	HPPE-500S-SUNN-DDD	See WS#17	378	
Former Shack 79 and 80	01 through 03	HPPE-SH79-SUNN-DDD /	See WS#17	42	

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SAP WS #18.4: Sampling Locations and Methods / SOP Requirements Table – Existing Building Surveys

Existing Building Number	Survey Unit Number (Range) *[NN]*	Type		Sample ID Number	Analytical Group	Sampling SOP Reference
404	01-43	Static Reading	SR	HPPE-404-SUNN-SR-DDD	See WS#17	WS#21
		Swipe	SW	HPPE-404-SUNN-SW-DDD	See WS#17	
414	01-20	Static Reading	SR	HPPE-414-SUNN-SR-DDD	See WS#17	
		Swipe	SW	HPPE-414-SUNN-SW-DDD	See WS#17	
521	01-08	Static Reading	SR	HPPE-521-SUNN-SR-DDD	See WS#17	
		Swipe	SW	HPPE-521-SUNN-SW-DDD	See WS#17	
810	01-29	Static Reading	SR	HPPE-810-SUNN-SR-DDD	See WS#17	
		Swipe	SW	HPPE-810-SUNN-SW-DDD	See WS#17	

Sample ID: Refer to SAP / Section 27.3.4 for details.
Sample ID Nomenclature: AABB-CCC-SUNN-TT-DDD
AA = facility
BB = site location
CCC = Building Number
SUNN = survey unit number
TT = Type
DDD = numeric sample digit (beginning with 001, in sequential order)

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SAP WS #18.5: Sampling Locations and Methods / SOP Requirements Table – Import Fill

- **Sample Location IDs**:
  - Locations have not been identified.
  - Sample ID nomenclature is listed on WS #27 (Section 27.3.5).
  - Detailed information for the source material will be recorded if applicable.
- **Methods:**
  - The required methods for import fill are listed on WS #30.
- **SOP Requirements**:
  - See WS #21 and WS #23.
  - Same sample procedures utilized by the excavated / trench soil samples.

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SAP WS #18.6: Sampling Locations and Methods / SOP Requirements Table – IDW

If project generated IDW requires laboratory(s) sampling analyses prior to disposal, then the samples will be labeled as follows:

HPPE-IDW-D####-MM-DDMMMYY

- HPPE = Site ID
- IDW = Investigation derived Waste
- D#### = Drum Number (0001 – 9999).
- MM = Matrix
  - LQ = Liquid
  - SO = Soil
  - SW = Soild Waste (Gloves, etc)
- DD = Sample Day / Date (numbers)(
- MMM = Month (Letters) – Jan. Feb, etc.
- YY = Year (2019 = 19, 2020 = 20, etc)

Methods of Analysis: Will be determined on the contents each drum.
Associated SOP: TBD

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SAP WS #19: ANALYTICAL STANDARD OPERATING PROCEDURE REQUIREMENTS TABLE

Matrix	Analytical Group	Analytical and Preparation Method	Sample Volume	Container	Preservation Requirements	Maximum Holding Time
Soil	Gamma Isotopes	Preparation and Analysis: EPA Method 901.1 MOD	250 to 400 grams	One 250-milliliter poly/glass jar, poly bag or tuna can	None	None
Soil	GFPC (^{90}Sr)	EPA Method 905.0	250 to 400 grams			
Soil	Alpha Isotopes (only if necessary)	U.S. Department of Energy Health and Safety Laboratory 300	250 to 400 grams			
Soil	Tritium (^{3}H)	EPA Method 906	-	-	-	-
Swipe	Alpha-Beta	N/A	Swipe of 100 cm^2 with 5.1 cm wipe	N/A	N/A	N/A

SAP WS #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE

Matrix	Analytical Group	No. of Primary Sampling Locations	No. of Field Duplicates	No. of MS/MSDs [2]	No. of Field Blanks	No. of Equipment Rinse Blanks	No. of Trip Blanks	Total No. of Samples to Laboratory
Soil (Radiological Screening Excavated Soil-Phase 1)	Gamma Isotopes	1,288 (estimated)	129	N/A - Not Required if tracers used by lab.	None [1]	None [1]	NA [2]	1,417 (estimated)
	GFPC Isotopes (10% of Gamma Isotopes)	129 (estimated)	13					142 (estimated)
	Alpha isotopes (only as necessary)	TBD [3]	TBD					TBD
Soil (Biased sampling)	Gamma Isotopes GFPC Isotopes (alpha isotopes only as necessary)	TBD—As needed to support field screening activities	None	N/A - Not Required if tracers used by lab.	None [1]	None [1]	NA [2]	TBD—As needed to support field screening activities
Soil (Borings Phase 2)	Gamma Isotopes	3,222 (estimated)	323	N/A - Not Required if tracers used by lab.	None [1]	None [1]	NA [2]	3,545 (estimated)
	GFPC Isotopes – Strontium 90 (10% of Gamma Isotopes)	323 (estimated)	33					356
	Alpha isotopes (only as necessary)	TBD	TBD					TBD
Soils (Former Building Surveys)	Gamma Isotopes	1,540	154	N/A - Not Required if tracers used by lab.	None [1]	None [1]	NA [2]	1,694
	GFPC Isotopes	154	16	None	None	None	None	170
	Alpha Isotopes (only as necessary)	TBD	TBD	None	None	None	None	TBD

SAP WS #20: FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE (continued)

Notes: [1] Field Blanks and Equipment blanks are only required if non-disposable sampling equipment is used in the collection of samples. This project plans on utilizing disposable sampling equipment.

[2] Trip Blanks are not required for the RAD Chem analyses. MS/MSDs are not required for this project, the laboratory will sue tracers to meet the precision portion of the DQO's.

[3] As Needed, based on Gamma field Screening (WS #17)

MS = matrix spike
MSD = matrix spike duplicate
N/A = not applicable
TBD = to be determined

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SAP WS #21: PROJECT SAMPLING STANDARD OPERATING PROCEDURE REFERENCES TABLE

21.1 SAMPLE COLLECTION METHODS AND PROCEDURES

The following subsections describe the sampling methods and procedures that may be used for the sample collection during this project.

Procedure	SOP Reference / Author / Date
Soil Sample Collection	ENV-601 / ECC / 26-May-2010
	ENV-801 / ECC / 08-June-2008
Soil Characterization Classification	ENV-802 / ECC / 19-June-2008
Swipe Sample Collection	SOP-RP-104 / Perma-Fix / May 2014

Copies are located in Attachment 3

21.2 CALIBRATION

Portable survey instruments will be calibrated annually at a minimum, in accordance with American National Standards Institute (ANSI) N323a-1997 Radiation Protection Instrumentation Test and Calibration, Portable Survey Instruments (ANSI N323) (ANSI, 1997), or an applicable later version. Instruments will be removed from service on or before calibration due dates for recalibration. If ANSI N323 does not provide a standard method, the calibration facility should comply with the manufacturer's recommended method.

Refer to the RPP for details on equipment calibration

21.3 DAILY PERFORMANCE CHECKS

Before use of the portable survey instruments, calibration verification, physical inspection, battery check, and source-response check will be performed in accordance with:

Procedure	SOP Reference / Authro / Date
Count Rate Instruments	SOP-RP-108 / Perma-Fix / May-2014
Dose Rate Instruments	SOP-RP-109 / Perma-Fix / May-2014

Copies are located in Attachment 3

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SAP WS #22: FIELD EQUIPMENT CALIBRATION, MAINTENANCE, TESTING, AND INSPECTION TABLE

Field Equipment	Calibration Verification Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Ludlum 3030 / Alpha Beta Sample Counter	-	-	-	-	RSO or Rad Tech	RP-108. RP-109
Ludlum 43-47 / Gas Proportional Counter	-	-	-	-	RSO or Rad Tech	RP-108. RP-109
Ludlum 43-68 / Gas Proportional Counter	-	--	-	-	RSO or Rad Tech	RP-108. RP-109
Ludlum 43-96 / Alpha Beta Scintillation	-	-	-	-	RSO or Rad Tech	RP-108. RP-109
Radiation Safety and Control Services – Position-Sensitive Proportional Counter	-	--	-	-	RSO or Red Tech	RP-108. RP-109

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SAP WS #23: ANALYTICAL STANDARD OPERATING PROCEDURE REFERENCES TABLE

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance from DoD QSM? (Y/N)
ST-RC-0003	Drying and Grinding of Soil and Solid Samples for Radiochemistry Analysis	Preparation	Soil	NA	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0025	Preparation of Samples for Gamma Spectroscopy, Rev 17, 12/19/17	Preparation	Soil	Gamma Spectrometer	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0030	The determination of tritium in water (and other fluids), soil, silica gels, and graphite (Rev 22, 09/23/2019)	Preparation	Soil	Liquid Scintillation	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0050	Aqueous and Soil Sample Preparation for Strontium-89, Strontium-90 and Total Strontium, 11/20/18	Preparation	Soil	Gas Flow Proportional Counter	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0058	Sample Preparation for Strontium 89, Strontium 90, and Total Strontium using Extraction Chromatography (03/31/2021)	Preparation	Soil	Sample Preparation for GFPC	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0100	Actinide Coprecipitation, 1/29/18	Definitive	Soil/Water	Alpha Spectrometer	Eurofins Test America, St. Louis Laboratory	N
ST-RD-0102	Gammavision® Analysis, Rev 19, 11/12/2019	Definitive	Solids Gamma Isotopes (^{226}Ra and ^{137}Cs)	Gamma Spectrometer	Eurofins Test America, St. Louis Laboratory	N
ST-RD-0210	Alpha Spectroscopy Analysis, 9/9/18	Definitive	Soil/Water	Alpha Spectrometer	Eurofins Test America, St. Louis Laboratory	N

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Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance from DoD QSM? (Y/N)
ST-RC-0242	Isotopic Thorium, Plutonium, and Uranium in Various Matrices by Eichrom separation resins, Rev 22, 09/23/2019	Definitive	Soil	Alpha Spectrometer	Eurofins Test America, St. Louis Laboratory	N
ST-RC-0301	Radium Isotopes by Alpha Spectrometry, 2/9/18	Definitive	Soil/Water	Alpha Spectrometer	Eurofins Test America, St. Louis Laboratory	N
ST-RD-0302	Liquid Scintillation Counter Analysis	Definitive	Soil	Liquid Scintillation	Eurofins Test America, St. Louis Laboratory	N
ST-RD-0403	Low Background Gas Flow Proportional Counting System Analysis, 12/15/17	Definitive	Solids, ^{90}Sr	Gas Flow Proportional Counter	Eurofins Test America, St. Louis Laboratory	N
Back Fill Soil (Offsite Source)						
DV-GC-0010	Gasoline Range Organics (GRO) by GC/FID, SW846 8015, Rev 15, 06/04/2020	Definitive	Soils	GC/FID	Eurofins Test America, Denver Laboratory	N
DV-GC-0020	Chlorinated Pesticides [EPA 8081A & 8081B], Rev 16, 09/19/2020	Definitive	Soils	GC	Eurofins Test America, Denver Laboratory	N
DV-GC-0021	Polychlorinated Biphenyls (PCB's) by GC/ECD (SW 846 Methods 8082 and 8082A), Rev 16, 03/03/2020	Definitive	Soils	GC/ECD	Eurofins Test America, Denver Laboratory	N
DV-GC-0027	Diesel and Residual Range Organics (DRO and RRO) by GC/FID [SW846 Methods 8015 and State Methods], Rev 10, 03/03/2020.	Definitive	Soils	GC/FID	Eurofins Test America, Denver Laboratory	N

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DV-MS-0010	Determination of Volatile Organics by GC/MS [8260B/C/D and 624./624.1], Rev 26, 03/13/2020	Definitive	Soils	GC/MS	Eurofins Test America, Denver Laboratory	N
33.308.8	STANDARD OPERATING PROCEDURE FOR DETERMINATION OF ASBESTOS CONTENT OF SERPENTINE AGGREGATE BY POLARIZED LIGHT MICROSCOPY and POINT COUNTING	Definitive	Soils	PLM	NVL Labs, Seattle WA	N
DV-MS-0011	GC/MS analysis Based on Methods 8270C and 625/625.1, Rev 16, 03/04/2020	Definitive	Soils	GC/MS	Eurofins Test America, Denver Laboratory	N
DV-MT-0016	Mercury in Solids by Cold Vapor Atomic Absorption	Definitive	Soils	CV-AA	Eurofins Test America, Denve Laboratory	N
DV-MT-0022	Inductively Coupled Plasma Mass Spectrometry for Trace Elements Analysis by SW-846 Method 6020A/B, Rev 10, 03/03/2020	Definitive	Soils	ICP/MS	Eurofins Test America, Denver Laboratory	N

Notes:

Attachment 2 provides SOPs.

Attachment 3 provides laboratory gamma spectroscopy library and current certification audit and MAPEP results for Eurofins Test America St. Louis laboratory.

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SAP WS #24: ANALYTICAL INSTRUMENT CALIBRATION TABLES

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SAP WS #24.1: Analytical Instrument Calibration Table - Laboratory

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Gamma Spectrometer	Initial Calibration (ICAL). (Energy, efficiency and Full Width at Half Maximum [FWHM] peak resolution)	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings.	The energy difference should be within 0.05% for all calibration points or within 0.2 keV. Peak energy difference is within 0.1 keV of reference energy for all points. Peak FWHM < 3 keV at 1,332 keV. The efficiency difference should be within 8% of the true value for each point unless T.C.C. calibration is performed.	Correct problem, then repeat ICAL.	Analyst / Supervisor	ST-RD-0102
Gamma Spectrometer	Initial Calibration Verification (ICV)	After ICAL for energy/efficiency and prior to analysis of samples.	Observed peaks of second source standard fall within ± 10% of ICAL value relative to the true value.	Verify second source standard and repeat ICV to check for errors. If that fails, identify and correct problem and repeat ICV or ICAL and ICV as appropriate.	Analyst / Supervisor	ST-RD-0102
Gamma Spectrometer	Continuing Calibration Verification (CCV) Daily Check	Daily or prior to use. When working with long count times or batch sequences that run more than a day, CCV is performed at the beginning and end of each analytical batch, as long as it is not longer than a week.	Energy: ± 0.5 keV at 60 keV; ± 0.75 keV at 1,332 keV FWHM: ± 1.2x at 60 keV; ±1.8x at 662 keV; ± 2.3x at 1,332 keV Activity Difference: % difference between the source activity and the reported activity ± 5%	Correct problem, rerun CCV. If CCV rerun fails, repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst / Supervisor	ST-RD-0102

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Gamma Spectrometer	Background Subtraction Count (BSC) Measurement (Long count for subtracting background from blanks or test sources)	Immediately after ICAL and then performed on at least a monthly basis.	Background count rate of the entire spectrum with ± 3σ of the average.	Recount and check control chart for trends. Determine cause, correct problem, re-establish BSC. If background activity has changed, then re-establish BSC and reanalyze or qualify all impacted samples since last acceptable BSC.	Analyst / Supervisor	ST-RD-0102
Gamma Spectrometer	Instrument Contamination Check (ICC) (Short count for controlling gross contamination)	Daily or when working with long count times before and after each analytical batch. Check after counting high activity samples.	No extraneous peaks identified (i.e., no new peaks in the short background spectrum compared to previous spectra); Background count rate of the entire spectrum with ± 3σ of the average.	Recount the background. If still out of control, then locate and correct problem; reanalyze or qualify all impacted samples since last acceptable ICC. If background activity has changed, then re-establish BSC and reanalyze samples.	Analyst / Supervisor	ST-RD-0102
Alpha Spectrometer	ICAL (Energy, efficiency, and FWHM peak resolution)	Prior to initial use, following repair or loss of control and upon incorporation of new or changed instrument settings.	3 isotopes within energy range of 3-6 MeV energy vs. channel slope equation < 15 keV per channel. FWHM < 100 keV for each peak used for calibration. Final peak energy within 20 keV of reference energy minimum of 3,000 net counts in each peak.	Correct problem, then repeat ICAL.	Analyst / Supervisor	ST-RD-0210

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Alpha Spectrometer	ICV	After ICAL.	The Full Width Half Maximum (FWHM) ≤ 100 keV for each peak. Each peak must fall within ± 20 keV of corresponding calibration peaks in the initial calibration. Minimum 2,000 net counts. Efficiency within 95% - 105% of ICAL efficiency value .	Repeat ICV to check for error. If that fails, identify and correct problem and repeat ICV or ICAL and ICV, as appropriate.	Analyst / Supervisor	ST-RD-0210
Alpha Spectrometer	CCV (Pulser check)	Pulser verification daily, prior to analysis of samples.	Gross counts within 5% of the average (20-point minimum). FWHM within 10-20 keV. Energy within ± 40 keV of the average (20-point minimum).	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Analyst / Supervisor	ST-RD-0210
Alpha Spectrometer	CCV (Check source)	Monthly source check verification prior to analysis of samples.	FWHM ≤ 100 keV Each peak within ± 30 keV of corresponding calibration peaks in initial energy calibration. Minimum 2,000 net counts. Efficiency within 95% - 105% of ICAL value.	Recount and check control chart for trends. Determine cause, correct problem, and repeat CCV and all associated samples since last successful CCV.	Analyst / Supervisor	ST-RD-0210
Alpha Spectrometer	BSC Measurement	Prior to initial use or after ICAL and monthly.	Use a statistical test to determine a change in the background count rate value.	Check control chart for trends and recount. Determine cause, correct problem, re-establish BSC. If background activity has changed, then re-establish BSC and reanalyze all impacted samples since last acceptable BSC.	Analyst / Supervisor	ST-RD-0210

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference
Alpha Spectrometer	ICC	Performed weekly, at minimum, and after counting high activity samples.	Blank ≤ 3 for blank subtracted (net) activity in all region of influence.	Check control chart for trends and recount. Determine cause and correct problem. If background activity has changed, then re-establish BSC and reanalyze all infected samples.	Analyst / Supervisor	ST-RD-0210
Gas Flow Proportional Counter	ICALV (separate plateaus determined for alpha and beta activity)	Prior to initial use and after loss of control.	Slope of the plateau less than 5% over a range of 100V.	Correct problem, then repeat ICALV.	Analyst / Supervisor	ST-RD-0403
Gas Flow Proportional Counter	Initial Calibration - Efficiency (ICALE)	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	Verify manufacturer's specifications for detector efficiency for both alpha and beta counting modes using electroplated sources.	Correct problem, then repeat ICALE.	Analyst / Supervisor	ST-RD-0403
Gas Flow Proportional Counter	ICAL – Cross-talk Factors	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	Verify manufacturer's specifications for cross-talk in alpha and beta channels.	Correct problem, then repeat ICALCT.	Analyst / Supervisor	ST-RD-0403

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Gas Flow Proportional Counter	ICAL – Self-Absorption Curve	Prior to initial use, after loss of control, and upon incorporation of new or changed instrument settings.	For each radionuclide of interest (or isotope with similar energy profile), establish mathematical function (curve) of detector efficiency vs. source mass loading. Best fit of data with coefficient of determination (r2) ≥ 0.9.	Correct problem, then repeat ICALSA.	Analyst / Supervisor	ST-RD-0403
Gas Flow Proportional Counter	IECV - Efficiency Calibration Verification	After ICALE for alpha and beta and prior to analysis of samples.	Individual points within ± 30% of true value, average of points within ± 10% of ICAL value.	Correct problem and verify second source standard. Rerun IECV. If that fails, correct problem and repeat ICALE.	Analyst / Supervisor	ST-RD-0403
Gas Flow Proportional Counter	CCV	After a counting gas change and daily for short test-source counting intervals.	Within tolerance or control chart limits ± 3% or 3σ of the mean.	Correct problem, rerun calibration verification. If that fails, then repeat ICALE. Reanalyze all samples since the last successful calibration verification.	Analyst / Supervisor	ST-RD-0403
Liquid Scintillation	ICAL	Prior to use	$r^2 > 0.99$ / or $r > 0.995$	Re-run ICAL	Analyst / Supervisor	ST-RD-0302
Liquid Scintillation	ICV	Following ICAL	+/- 10% of known value	Re-analysis of ICV	Analyst / Supervisor	ST-RD-0302
Liquid Scintillation	CCAL	Each day that the measured nuclides are performed	Within Lab QC limits	If CCAL fails - Re-run. If re-run fails, run is terminated.	Analyst / Supervisor	ST-RD-0302

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SAP WS #24.2: Analytical Instrument Calibration Table – Field Analyses and measurements

The field analyses and measurements will be performed by the following instruments. Please refer to the RPP for the calibration procedures.

- Ludlum 3030 / Alpha-Beta Sample Counter
- Ludlum 43-37 / Gas Proportional Detectors
- Ludlum 43-68 / Gas Proportional Detectors
- Ludlum 43-96 / Alpha-Beta Scintillation Detectors
- Radiation Safety and Control Services – Surface Contamination Monitor / Position-sensitive Proportional Counters

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SAP WS #24.3: ANALYTICAL INSTRUMENT CALIBRATION TABLE – ORGANIC AND INORGANIC ANALYSES

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
GC/MS volatile and semivolatile organic analyte	Check of mass spectral ion intensities (tuning procedure EPA Method 8260 and 8270)	Prior to ICAL and prior to each 12-hour period of sample analysis	Must meet the EPA method tune requirements specific ion abundance criteria of bromofluorobenzene or decafluorotriphenylphosphine before samples are analyzed	If necessary, perform maintenance, retune instrument, and verify the tune acceptability	Analyst / Supervisor	
	ICAL: Minimum five levels for linear and six levels for quadratic	ICAL prior to sample analysis	Each analyte must meet one of the following: Option 1: relative standard deviation for each analyte ≤ 15% Option 2: linear least squares regression correlation $r^2 \geq 0.99$ Option 3: nonlinear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$	If necessary, perform maintenance and Retune instrument repeat ICAL		
	Second-source ICV	Once after each ICAL	Analytes within ± 20% of true value	Correct problem and rerun ICV If that fails, repeat ICAL		
	Retention time (RT) window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed On days when ICAL is not performed, the initial CCV is used	Not applicable		
	Evaluation of relative RT	With each sample	Relative RT of each target analyte within ± 0.06 relative RT units	Determine problem, then rerun ICAL		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
	CCV	Daily before sample analysis; after every 12 hours of analysis time and at the end of the analytical batch run	Reported analytes and surrogates within ± 20% of true value Reported analytes and surrogates within ±50% for end of analytical batch CCV	Recalibrate, and reanalyze affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs If both pass, samples may be reported without reanalysis If either fails, take corrective action(s) and recalibrate; then reanalyze affected samples since the last acceptable CCV		
	Performance Check (EPA Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples	Degradation ≤ 20% for Dichlorodiphenyl trichloroethane. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2	Correct problem, then repeat performance checks	Analyst/ Supervisor	
	Internal standards (ISs) verification	Every field sample, standard, and QC sample	RT ± 10 seconds from RT of the midpoint standard in the ICAL; Extracted Ion Current Profile area within -50% to +100% of ICAL midpoint standard	Inspect detector and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory		
ICP/MS	Linear Dynamic Range or High-Level Check Standard	At initial set-up and checked every 6 months with a high standard at the upper limit of the range	Within ± 10% of true value.	Dilute samples within the calibration range or reestablish/verify the Linear Dynamic Range.		

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
	Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height	Correct problem and repeat tune		
	ICAL for All Analytes	Daily ICAL prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.99$	Correct problem and repeat ICAL		
	ICV	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL		
	CCV	After every 10 field samples and at the end of the analysis sequence	All reported analytes within ± 10% of the true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails or if two consecutive CCVs cannot be run, then perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.		
ICP/MS	Low-Level Calibration Check Standard	Daily. Low-Level Calibration Check Standard should be ≤to the LOQ.	All reported analytes within ± 20% of the true value	Correct problem and repeat ICAL	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
	IS	Every field sample, standard and QC sample.	IS intensity in the samples within 30-120% of intensity of the IS in the ICAL blank.	If recoveries are acceptable for QC samples, but not field samples, the field samples may be considered to suffer from a matrix effect. Reanalyze sample at 5-fold dilutions until criteria is met. For failed QC samples, correct problem and rerun all associated failed field samples.		
Cold Vapor	ICAL Minimum five standards and calibration blank	Daily ICAL prior to sample analysis	If more than one calibration standard is used, $r^2 \geq 0.99$	Correct problem, then repeat ICAL	Analyst/ Supervisor	
	Second-source ICV	Once after each ICAL, prior to beginning a sample run	Analyte(s) within ± 10% of true value	Correct problem Rerun ICV If that fails, rerun ICAL		
	CCV	After every 10 field samples and at the end of the analysis sequence	Within ± 10% of true value	Recalibrate, and reanalyze affected samples since the last acceptable CCV; or immediately analyze two additional consecutive CCVs If both pass, samples may be reported without reanalysis If either fails, take corrective action(s) and recalibrate; then		

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Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
				reanalyze affected samples since the last acceptable CCV		
	Initial and continuing calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > LOD	Correct problem and repeat ICAL Samples following the last acceptable calibration blank must be reanalyzed		
GC/FID or GC/ECD	Minimum five-point ICAL for analytes	ICAL prior to sample analysis	One of the options below: Option 1: relative standard deviation for each analyte ≤ 20% Option 2: linear least squares regression: $r^2 \geq 0.995$ Option 3: nonlinear regression: coefficient of determination $r^2 \geq 0.99$ (six points shall be used for second order; seven points shall be used for third order)	Correct the problem; then repeat ICAL.	Analyst/ Supervisor	
	Endirn/ Dichlorodiphenyl trichloroethane breakdown check (8081 only)	Before sample analysis and at the beginning of each 12-hour shift	Degradation of Dichlorodiphenyl trichloroethane and Endrin must each be ≤ 15%	Correct the problem; then repeat breakdown check		
	ICV	Once after each ICAL, analysis of a second-source standard prior to sample analysis	Project analytes within established RT windows GC methods: project analytes within ± 20% of expected value from the ICAL	If necessary: Perform instrument maintenance, then repeat ICV. If that fails, repeat ICAL.		
	RT window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical sequence	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed on days when ICAL is not performed, the initial CCV is used	Perform instrument maintenance if necessary; adjust windows, then re-evaluate analyte identification		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for CA	SOP Reference[1]
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for pesticides multi-component analytes (i.e. toxaphene, chlordane), which are only required before sample analysis	Project analytes within established RT windows Project analytes within ± 20% of expected value from the ICAL	Determine the source of the problem and correct, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze samples since the last successful calibration verification.		
Polarized Light Microscope	Microscope alignments	Before each use	Microscope aligned using Anthophyllite fibers mounted in 1.605 RI liquid to ensure proper alignment of polarizer and analyzer. The polarizer and analyzer must be set at 90 degree to one another.	Perform Microscope maintenance	Analyst/ Supervisor	

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SAP WS #25: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION

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SAP WS #25-1: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table / RAD chem

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Gamma Spectrometer	Liquid Nitrogen fill	Physical check	Physical check	Weekly	Acceptable background	• Recalibrate • Instrument maintenance • Consult with Technical Director	Analyst/Supervisor	
Alpha Spectrometer	1. Vacuum Pump Oil replacement 2. Filter cleaning on the air intake of the instrument cabinet	Physical check	Physical check	1) Semi Annually 2) Quarterly	Acceptable background and calibration efficiencies	• Recalibrate • Instrument maintenance • Consult with Technical Director	Analyst/Supervisor	
Gas Flow Proportional Counter	Sample Shelf Cleaning	Physical check	Physical check	Weekly	N/A	N/A	Analyst/Supervisor	
Liquid Scintillation	-	-	-	-	-	--	-	

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SAP WS #25-2: ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTION TABLE / Organic & inorganic

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC	Injection port maintenance	Preventative maintenance	Change septum, clean injection port, clip column	Daily	CCV passes criteria (Worksheet #24)	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst / Department Manager	WS #23
GC	Trap	CCV response dropping, sample foamed over	Change trap	When responses start to drop or after foam over samples	CCV passes criteria	Re-bake trap, replace trap, reanalyze CCV, recalibrate	Analyst / Department Manager	WS #23
GC/MS	Injection port maintenance	Preventative maintenance	Change septum, clean injection port, clip column	Daily	Tune and CCV pass criteria	Re-inspect injector port, cut additional column, reanalyze CCV, recalibrate instrument	Analyst / Department Manager	WS #23
GC/MS	Trap	CCV response dropping, sample foamed over	Change trap	When responses start to drop or after foam over samples	Tune and CCV passes criteria	Re-bake trap, replace trap, reanalyze CCV, recalibrate	Analyst / Department Manager	WS #23

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Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Detector maintenance	Column change, unable to tune instrument	Clean detector, change pump oil	When responses drop and tunes start to fail	Tune passes, air and water are not present in the scan	Disassemble detector and check parts, check heating element, reanalyze tune	Analyst / Department Manager	WS #23
ICP/MS	Injector Maintenance	Preventative maintenance	Change injector and torch	Daily to every other day	Intensity of 1 part per million manganese standard within criteria	Replace, investigate injector, reanalyze	Analyst / Department Manager	WS #23
ICP/MS	Pump maintenance	Preventative maintenance	Replace pump windings	Daily	Monitor IS counts for variation	Replace windings, recalibrate and reanalyze	Analyst / Department Manager	WS #23
Polarized Light Microscope	Check microscope alignments	Physical check	Physical check	Daily	Microscope aligned using Anthophyllite fibers mounted in 1.605 RI liquid to ensure proper alignment of polarizer and analyzer. The polarizer and analyzer must be set at 90 degree to one another	Instrument maintenance Consult with Technical Director	Analyst / Department Manager	WS #23

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SAP WS #26: SAMPLE HANDLING SYSTEM

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): EIP Field Technician or Project Chemist
Sample Packaging (Personnel/Organization): EIP Field Technician or Project Chemist
Coordination of Shipment (Personnel/Organization): EIP Field Technician or Project Chemist
Type of Shipment/Carrier: Lab Courier, UPS, or FedEx
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Laboratory Sample Receiving and PM
Sample Custody and Storage (Personnel/Organization): Laboratory Sample Receiving and PM
Sample Preparation (Personnel/Organization): Laboratory Sample Receiving and PM
Sample Determinative Analysis (Personnel/Organization): Laboratory Sample Receiving and PM
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): For the samples shipped to laboratory for off-site analysis, the samples will be shipped the same day as collection if possible, if not possible to ship the same day; storage on site in refrigerator or ice-packed cooler in locked building
Lab Sample Storage (No. of days from sample collection): Minimum six months after results are received by EIP Project Chemist
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 30 days
Biological Sample Storage (No. of days from sample collection): Not Applicable to this project
SAMPLE DISPOSAL
Personnel/Organization: Laboratory Sample Receiving and PM
Number of Days from Analysis: 7-Months – Contact EIP prior to action Laboratory samples and remaining sample volume will be returned under chain-of-custody for archiving to: • Company: EIP • Attention: Project Chemist • Address: TBD

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SAP WS #27: SAMPLE CUSTODY REQUIREMENTS TABLE

27.1 SAMPLE CUSTODY AND DOCUMENTATION

Sampling information will be recorded on a Chain of Custody Form, in a permanently bound field logbook, and in electronic field forms. All entries will be legible and recorded in indelible ink.

27.2 FIELD SAMPLE CUSTODY PROCEDURES

Field sample custody procedures include sample collection, packaging, shipment, and delivery to the laboratory(s). Custody of field samples will be maintained, and custody transfer will be documented from the time of sample collection through receipt of samples at the analytical laboratory(s) using chain-of-custody and custody seal procedures. The Sample Management Coordinator or qualified designee will fulfill these requirements. Enhanced sample custody will include the following:

- Each sample will be collected into the containers presented in WS #19.
- Sample custody seal will be applied to each sample container.
- A sample custody bag will be used to group samples together into like TUs/SUs.

Each sample will be considered to be in the sampler's custody if one of the following occurs:

- Sample is in the person's physical possession.
- Sample is in view of the person after that person has taken possession.
- Sample is secured so that no one can tamper with the sample.
- Sample is secured in an area that is restricted to authorized personnel.

Samples will be shipped directly from the field to the analytical laboratory(s). Samples will be packaged and shipped for off-site analysis in accordance with SOP packaging and shipping procedures for low-concentration samples (SAP WS #21).

27.3 SAMPLE LABELING

The former trenches will be excavated and characterized in "batches" that will be given new unique identifiers at the time of excavation by the geologist or radiation technician. Excavated material representing the backfill material from former trenches will use the following nomenclature format: Each sample container will be labeled with the following, at minimum:

- Sample ID number
- Sample collection date (DD-MMM-YYYY)
    - DD and YYYY are numerical, MMM is letters (Jan, Feb, etc)
    - Example – 01-Jan-2019

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- Time of collection (24-hour clock)
- Sampler's initials
- Analyses to be performed
- Preservation (if any)
- Location (i.e., site name)

27.3.1 Phase 1 TU Soils:

Sample identifications (IDs) from the Phase 1 trench confirmation investigation will be identified using the following format:

AABB-CCC-NNNA-DDD

Where: AA = facility ("**HP**" for Hunters Point will be used in this WP)
BB = site location (Parcel E = **PE**)
CCC = ESU or SFU
NNN = former TU number
A = Unique Batch ID, alpha-ID only (beginning with A, in sequential order)
DDD = numeric sample digit (beginning with 01, in sequential order)

For example, the first soil sample collected from the first "batch" of backfill TU material excavated from the former TU 34 will be identified as follows:

- Site Sample: **HPPE-ESU-154A-001**
- Field Duplicate: **HPPE-ESU-154A-001D** (will use the same number but add a "D")
- Re-Sampling **HPPE-ESU-154A-001-RE**

In this example,

- "HPPE" identifies Hunters Point Parcel E,
- "ESU" identifies excavation soil unit,
  - SFU – Sidewall Floor Unit
- "154" identifies the unit as being excavated from the former TU 154,
- "A" represents the first batch (B for second batch, etc) created from excavating this former TU, and
- "001" identifies the first sample

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27.3.2 Phase 2 TU Soils:

Sample IDs from the Phase 2 soil trench confirmation investigation will be identified using the following format:

AABB-CCC-T-SB##-DDDD

Where: AA = facility ("HP" for Hunters Point will be used in this WP)
BB = site location ("PE" for Parcel E will be used in this WP)
T = Type (C = center of Trench / S = sidewall)
CCC = TU number
SB##= soil boring number
DDDD = four-digit top of sample interval in feet below ground surface (each sample interval will be approximately 6 inches)

For example, the tenth soil boring sample collected at the surface from the TU 154 in Parcel E will be identified as follows:

HPPE-154-C-SB01-0005

In this example, "HPPE" identifies Hunters Point Parcel E, "154" identifies the former TU 154, "C" represents Center of trench soil boring, "SB10" identifies soil boring location number 10, and "0005" represents the first sample (surface) from 0 to 0.5 ft depth.

27.3.3 Former Building Sites

Sample IDs for samples collected at the Former Building / SU confirmation investigation will be identified using the following format:

AABB-CCCC-SUNN-DDD

Where: AA = facility ("**HP**" for Hunters Point will be used)
BB = site location ("**PE**" for Parcel E will be used)
CCCC = building site name
SUNN = survey unit number
DDD = numeric sample digit (beginning with 001, in sequential order)

For example, the second soil sample collected from former Building 520 in Survey Unit 2 will be identified as follows:

HPPE-520-SU02-002

In this example, "520" identifies former Building 520, "SU02" identifies the unit as being Survey Unit 2, and "002" identifies the second sample.

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27.3.4 Existing Building Site

Sample IDs for samples collected at the Former Building / SU confirmation investigation will be identified using the following format:

AABB-CCC-SUNN-TT-DDD

Where: AA = facility (HP for Hunters Point will be used)
BB = site location (PE for Parcel E will be used)
CCC = building number (404, 414, 521, or 810)
SUNN = survey unit number
TT = Type
- SR = Static Reading
- SW = Swipe

DDD = numeric sample digit (beginning with 001, in sequential order)

For example, the first Static Reading sample collected from former Building 404 in Survey Unit 1 will be identified as follows:

HPPE-404-SU01-SR-001

27.3.5 Import Fill

If import fill is required, the following Sample IDs nomenclature is required:

AABB-IF-S###-01-

Where: AA = facility (HP for Hunters Point will be used)
BB = site location (PE for Parcel E will be used)
IF = Import Fill
S### = Source Number (S001, S002, etc)
= Sample location Number#

Example: HPPE-IF-S001-01

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27.4 CHAIN-OF-CUSTODY

Attachment 1 includes an example of the electronic Chain-of-Custody (COC) Form that will be completed out via an electronic tablet program by EIP for samples submitted to Test America, this will the primary source of data recording. If an electronic version is not able to be utilized, the same TA COC will be printed for use as a hardcopy. In addition to providing a custody exchange record for the samples, the COC Form serves as a formal request for sample analyses. The COC Form will be completed, signed, and distributed as follows:

- One copy retained by the sample coordinator (this may be an electronic copy - electronic copies will be stored on the project SharePoint folder).
- Electronic copies will printed to PDF prior to sample shipment.
- The PDF copies will be emailed to the project folder
- The original sent to the analytical laboratory(s) with the sample shipment.

Samples will be shipped directly to the laboratory(s) performing the analysis

Laboratory Receipt of COCs

After the laboratory(s) receives the samples, the Sample Custodian will inventory each shipment before signing for it and note discrepancies in the number of samples on the original COC Form, temperature of the cooler or broken samples. The Project Chemist will be notified immediately of problems identified with shipped samples. The Project Chemist will in turn notify the Project QC Manager or Program Chemist, and together they will determine the appropriate course of action. The Project Chemist will also notify the PM or CM if the project budget and schedule may be impacted.

The laboratory(s) will initiate an internal chain-of-custody that will track the sample within the various areas of the laboratory(s). The relinquishing signature of the Sample Custodian and the custody acceptance signature of the laboratory(s) personnel transfer custody of the sample. This procedure is followed each time a sample changes hands.

The laboratory(s) will archive the samples and maintain their custody as required by the contract or until further notification from the Project Chemist, at which time the samples will either be returned to the project for disposal or disposed by the laboratory(s).

27.5 FIELD LOGBOOKS

A permanently bound field logbook with consecutively numbered pages will be assigned to this project. Entries will be recorded in indelible ink to each field sampling crew. Corrections will be made following the procedure described in Section 27.6. At the end of each workday, the responsible

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sampler will sign the logbook pages and unused portions of a logbook page will be crossed-out, signed, and dated. The logbook will be the secondary source of data recording. Once the electronic COC has been completed, the field tech will manually record the sample information listed below.

At a minimum, the logbook will contain the following information:

- Project name and location (on the front page of the logbook)
- Date and time of collection for each sample
- Sample number
- Sample location (i.e., soil boring or sampling point)
- Sample type (i.e., soil, air, and water) – systematic or bias
- Composite or grab if collected; composite type (the number of grab samples)
- Depth of sample
- Weather information (e.g., rain, sunny, approximate temperature)
- Containers used and requested analyses

In the of the field logbook (graph paper portion or equivalent), the sampler will provide the following information:

- A map with sample locations (drawn or paste copy on the graph paper portion or equivalent). Each sample location must be clearly identified on the map. Several sample locations may be presented on one map; however, the page with the map must be referred on each of the individual sample pages.
- Field analyses performed, including results, instrument checks, problems, and calibration records for field instruments.
- Descriptions of deviations from this SAP.
- Problems encountered and corrective action taken.
- Identification of field QC samples (Field Duplicates or Client Split Sample)

Attachment 1 provides example of the field logbook and the associated field forms.

27.6 DOCUMENT CORRECTIONS

Hardcopy changes or corrections on project documentation will be made by crossing out the item with a single line, initialing by the person performing the correction, and dating the correction. The original item will remain legible beneath the cross out. The new information will be written above the crossed-out item. Corrections will be written clearly and legibly with indelible ink.

Electronic changes will be submitted in email format to the affected personnel and saved in PDF format with the relevant changes brief notes to the COC folder in the project Sharedrive files. If possible, the original document will be included as a screen shot within the email or as an attachment. The email subject line will the pertinent information relevant to the change (Project, COC number, sample ID, etc)

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SAP WS #28: LABORATORY QUALITY CONTROL SAMPLES TABLES

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SAP WS #28.1: Laboratory Quality Control Samples Table (Gamma Isotopes)

Matrix	**Soil**
Analytical Group	**Gamma Radionuclides**
Analytical Method / SOP Reference:	**EPA 901.1M/SOP ST-RD-0102**

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Method Blank (MB)	One per analytical batch	|ZBlank |≤3 for blank subtracted (net) activity in radionuclides of interest (MARLAP 18.4.1 [EPA et al., 2004])	Recount the blank to confirm results, unless sample results are > five times the blank activity	Laboratory Manager/ Analyst	A means of assessing the existence and magnitude of contamination introduced via the analytical process	No analytes detected > two times the blank combined standard uncertainty. Blank result must not otherwise affect sample results
		In-house control limits of ± 3 σ of the mean	Inspect MB control chart for indication of significant bias			
			If required, re-prep and reanalyze MB and samples processed with the contaminated blank			
LCS	One per analytical batch	|ZLCS |≤3. Investigate recurrent results with |ZLCS| greater than or equal to (≥) 2 (MARLAP 18.4.3)	Recount the LCS to confirm results	Laboratory Manager/ Analyst	Accuracy	In-house control chart limits of ±3σ of the mean
		Or Use in-house control chart limits of ±3σ of the mean	Inspect LCS control chart for indication of significant bias			In-house control limits may not fall more than 25% from the known LCS value

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		In-house control limits may not fall more than 25% from the known LCS value	If required, re-prep and reanalyze the LCS and associated samples			
Sample Duplicate	One per analytical batch	|ZDup | ≤3. Investigate recurrent results with |ZDup|≥2 (MARLAP 18.4.1)	Check for lab error. Examine the project-specific requirements	Laboratory Manager/ Analyst	Precision	The DER between the sample and the duplicate is <3; or the RPD is <25%
		Or the duplicate error ratio (DER) between the sample and the duplicate is <3; or the RPD is <25%	Contact the client as to additional measures to be taken			

*Precision for the Gamma analyses utilizes the sample duplicate analyses. Eurofins Test America does not analyze an LCSD, MS/MSD or a tracer. The sample duplicate is the same aliquot analyzed on two different detectors. Internal lab criteria precision is RPD < 40% or RER < 1.

SAP WS #28.2: Laboratory Quality Control Samples Table (Alpha Spectroscopy)

Matrix	**Soil**					
Analytical Group	**Radiological (alpha spectroscopy)**					
Analytical Method / SOP Reference	**DOE Method HASL-300 A-01-R / ST-RC-0242**					
QC Sample	**Frequency Number**	**Method / SOP QC Acceptable Limits**	**Corrective Action**	**Person(s) Responsible for CA**	**Data Quality Indicator**	**Measurement of Performance**
Method Blank	One per prep batch of 20 or fewer samples of similar matrix or one per day whichever comes first	No Analytes Detected > MDC	Correct Problem. If required, re-prep and re-analyze MB and all samples associated with contaminated blank.	Analyst / Supervisor.	Bias / Contamination	Same as Method / SOP QC acceptance limits.
LCS		Recovery Limits:	Identify problem; if not related to matrix interference, re-reanalyze	Analyst/	Accuracy / Bias	
			LCS and all associated batch samples.	Supervisor		
		238Pu: 80 to 127%				
		239/240Pu: 75 to 125% 238U: 75 to 125% 226Ra: 75 to 125% 232Th: 75 to 125%				

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Tracer	Per sample, blank, LCS, MS, MSD (MS not required when yield tracers are employed)	217At tracer: 30 to 110% 242 or 236Pu tracer: 30 to 110% 232U tracer: 30 to 110% 229Th tracer: 30 to 110%	Truncate tracers above 110% recovery to eliminate low biased results. Re-prepare and reanalyze sample if carrier is low (indicating high biased results) if there is activity in the sample above the reporting limit. No reanalysis if matrix interference is nonconformance during sample preparation.	Analyst/ Supervisor	Accuracy / Bias	

SAP WS #28.2: LABORATORY QUALITY CONTROL SAMPLES TABLE (ALPHA SPECTROSCOPY) – (continued)

Matrix:	**Soil**
Analytical Group	**Alpha Isotopes**
Analytical Method / SOP Reference	**DOE A-01-R /SOP ST-RD-0210**

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Sample Duplicate	One per analytical batch	|ZDup| ≤ 3. Investigate recurrent results with |ZDup| ≥ 2 (MARLAP 18.4.1) Or the DER between the sample and the duplicate is < 3; or the RPD is < 25%	Check for lab error. Examine the project-specific requirements Contact the client as to additional measures to be taken	Laboratory Manager / Analyst	Precision	RPD is 25% or less or DER ≤ 3

Notes: The laboratory limits will be used.

> = greater than
≥ = greater than or equal to
< = less than
≤ = less than or equal to
^{232}U = Uranium-232
CA = Corrective Action
DOE = United States Department of Energy
LCS = laboratory control sample
MDC = minimum detected concentration

SAP WS #28.3: Laboratory Quality Control Samples Table (Gas Flow Proportional Counting Isotopes)

Matrix	**Soil**					
Analytical Group	**Gas Flow Proportional Counting**					
Analytical Method / SOP Reference	**EPA 905 Mod / ST-RD-0403**					
QC Check	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for CA**	**Data Quality Indicator**	**Measurement Performance Criteria**
Method Blank	One per prep batch of 20 or fewer samples of similar matrix or one per day, whichever comes first	No analytes detected > MDC	Correct problem. If required, re-prepare and re-analyze method blank and all samples processed with the contaminated blank.	Laboratory Manager/ Analyst	Bias / Contamination	Same as Method / SOP QC acceptance limits
LCS		Recovery Limits: 75-125%	Identify problem; if not related to matrix interference, re-reanalyze LCS and all associated batch samples.		Accuracy / Bias	
LCSD		Relative Percent Difference ≤ 25% and or RER ≤ 1	Correct problem, then re-reanalyze all samples processed with the duplicate.		Precision	
Carriers (if used)	Per Sample, blank, LCS, MS/MSD (MS are not required when field tracers are employed)	Strontium and Yttrium Carriers: 40-110%.	Truncate Carriers above 100% recovery to eliminate low biased results. Re-prepare and reanalyze sample if carrier is low (indicating high biased results) if there is activity in the sample above the reporting limit. No reanalysis if matrix interference is nonconformance during sample preparation.		Accuracy / Bias	

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SAP WS #28.4: LABORATORY QUALITY CONTROL SAMPLES TABLE (Liquid Scintillation)

Matrix	**Soil**
Analytical Group	**Tritium (^3H)**
Analytical Method / SOP Reference	**EPA Method 906 / ST-RC-0302**

QC Sample	Frequency Number	Method / SOP QC Acceptable Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement of Performance
Method Blank	One per prep batch of 20 or fewer samples	No target analytes present above ½ RL	Blank contamination above the RL requires re-prep of batch unless all associated samples are < RL or greater than five times the amount detected in the method blank.	Analyst / Supervisor	Bias / Contamination	Same as Method / SOP QC acceptance limits.
LCS / LCSD		All control analytes must be within the specified control limits for accuracy (% Recovery) and precision (RPD/RER).	(high) – Samples that are non-detect may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS. RPD's: are re-prepped and re-analyzed. (low) – batch is re-prepped and re-analyzed.	Analyst	Accuracy / Bias	
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)		Analyte(s) should be within control limits for accuracy (% Recovery) and precision (RPD/RER).	MS/MSD Spike Recovery excursion may not necessarily warrant corrective action other than narration. If affected analyte activity in the original sample is greater than five times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to physically evident matrix interferences, then the data is reported with an NCM (the physical interferences must be described in the NCM). If there is no evidence of interference and the RPD/RER as well as spike recoveries are out outside limits, then sample re-prep and re-analysis are required.	Analyst	Accuracy / Bias	

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SAP WS #28.4: LABORATORY QUALITY CONTROL SAMPLES TABLE (Liquid Scintillation) (continued)

Field Duplicate		-	For the RPD/RER, one or both must be within acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a re-prep of the samples. If the sample(s) have a physical matrix issue (i.e. non-homogenous), then results can be reported with an NCM. If the sample(s) fail RPD/RER criteria after a re-prep and no matrix issue is observed, then sample(s) may be reported with client approval and narrated in an NCM. If the sample(s) associated to the batch have activity below the RL, then sample(s) may be reported with an NCM.	Analyst	Accuracy / Bias	
Tracer	-	Tracer recovery must be within specified limits.	Low Rec or High Rec: batch is re-prepped and re-analyzed.	Analyst	Accuracy / Bias	

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SAP WS #28.5: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 8015

Matrix: Soil

Total Petroleum Hydrocarbons (TPH) by Gas Chromatograph (GC)

EPA Method 8015

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
ISs (optional)	Every sample, spiked sample, standard, and MB	Areas within -50% to +100% of last calibration verification	Identify problem; if not related to matrix interference, then re-extract and reanalyze affected samples.	Laboratory Manager / Analyst	Quantitation	Areas within -50% to +100% of last calibration verification
MB	One per analytical batch (TPH as gasoline) and one per preparation batch (TPH as diesel)	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Determine the source of contamination, reanalyze blank, then re-extract and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit
LCS or LCS/LCSD	One LCS or LCS/LCSD pair per analytical/preparation batch	Control limits—Attachment 2	Re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Manager / Analyst	Precisions and Accuracy	Control limits—Attachment 2
MS/MSD for analytes	One MS/MSD pair per preparation batch Batch = 20 samples or less	Control limits—Attachment 2	Examine the project-specific DQOs. If the MS falls outside of DoD criteria, then evaluate matrix effects.	Laboratory Manager / Analyst	Precision and Accuracy	Control limits—Attachment 2

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Matrix: Soil

Total Petroleum Hydrocarbons (TPH) by Gas Chromatograph (GC)

EPA Method 8015

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Surrogate standards	Every sample, spiked sample, standard, and MB	Control limits—Attachment 2	For QC and field samples, correct problem then re-prep and reanalyze failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	Surrogate standards	Every sample, spiked sample, standard, and MB	Control limits—Attachment 2

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SAP WS #28.6: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 6020

Matrix: Solid
Inductively Coupled Plasma Metals
EPA Method 6020

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Calibration blank	Immediately after the ICV and after every CCV	No analytes detected > ½ LOQ < 1/10th the amount measured in any sample	Determine source of blank contamination and correct problem Re-prep and reanalyze calibration blank samples following the last acceptable calibration blank must be reanalyzed	Laboratory Manager / Analyst	Representativeness	No target analytes ≥ ½ LOQ
MB	One per digestion batch	No analytes detected > ½ LOQ < 1/10th the amount measured in any sample	Determine source of blank contamination If required, re-prep and reanalyze MB and samples processed with the contaminated blank	Laboratory Manager / Analyst	Representativeness	No target analytes ≥ ½ LOQ
Interference check solution (ICS)	After ICAL and prior to sample analysis	ICS-A—Absolute value of concentration for non-spiked analytes < ½ LOQ (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ±20% of true value	Terminate analysis, perform maintenance to correct the problem reanalyze ICS and affected samples	Laboratory Manager / Analyst	Accuracy	Within ±20% of expected value

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Matrix: Solid
Inductively Coupled Plasma Metals
EPA Method 6020

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
LCS or LCS/LCD pair if there is not enough sample for MS/MSD	One LCS or LCS/LCD pair per each preparation batch	Control limits—Attachment 2	Determine source of LCS failure and correct problem, then re-prep and reanalyze the LCS and samples in the associated preparatory batch if enough sample material is available	Laboratory Manager / Analyst	Precision and Accuracy	Control limits—Attachment 2
MS/MSD for analytes	One MS/MSD pair per preparation batch	Control limits—Attachment 2	Examine the project-specific DQOs If the MS falls outside of DoD criteria, evaluate matrix effects Perform post-digestion spike	Laboratory Manager / Analyst	Precision and Accuracy	Control limits—Attachment 2
Post-Digestion Spike	Perform if MS/MSD fails One per preparation batch (using the same sample as MS/MSD)	Control Limits—Recovery 80%-120%	Narrate the results	Laboratory Manager / Analyst	Precision and Accuracy	Control Limits—80%-120%
Dilution test	One per preparatory batch if MS/MSD fails Only applicable for samples with concentrations > 50 × LOQ (prior to dilution)	Five-fold dilution must agree within ±10% of the original measurement	Evaluate for matrix interference Use along with MS/MSD and post-digestion spike data to confirm matrix effects	Laboratory Manager / Analyst	Accuracy	10% Difference

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SAP WS #28.7: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 8270 / 8270-SIM

Matrix: Soil
Semivolatile Organic Compounds (SVOCs) and Polycyclic Aromatic Hydrocarbons (PAH's)
EPA Method 8270/8270-SIM

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Surrogate standards	Every sample, spiked sample, standard, and MB	Control limits—Attachment 2	For QC and field samples, correct problem then re-prep and reanalyze failed samples for failed surrogates in the associated preparatory batch, if enough sample material is available. If obvious chromatographic interference with surrogate is present, then reanalysis may not be necessary.	Laboratory Manager / Analyst	Accuracy	Control limits—Attachment 2
MB	One per analytical batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit	Correct problem; If required, re-prep and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit
MS/MSD	One MS/MSD per analytical/ preparation batch	Control limits—Attachment 2	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and associated batch samples.	Laboratory Manager / Analyst	Precision and Accuracy	Control limits—Attachment 2
LCS or LCS/LCSD	One per preparatory batch	Control limits—Attachment 2	Determine the source of the problem and correct, then re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed LCS analytes, if enough sample material is available.	Laboratory Manager / Analyst	Precision and Accuracy	Control limits—Attachment 2

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Matrix: Soil
Semivolatile Organic Compounds (SVOCs) and Polycyclic Aromatic Hydrocarbons (PAH's)
EPA Method 8270/8270-SIM

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparation batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit	Determine the source of blank contamination and correct problem. If required, re-prep and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit
MS/MSD for analytes	One MS/MSD pair per analytical/preparation batch	Control limits—Attachment 2	Examine the project-specific DQOs. Contact the client as to additional measures to be taken	Laboratory Manager / Analyst	Precisions and Accuracy	Control limits—Attachment 2
LCS or LCS/LCSD Containing Analytes Reported	One LCS or LCS/LCSD pair per analytical/preparation batch	Control limits—Attachment 2	Determine the source of the problem and correct, then re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Manager / Analyst	Precisions and Accuracy	Control limits—Attachment 2

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SAP WS #28.8: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 8081 / 8082

Matrix: Soil
Organochlorine Pesticides (Pest) and Polychlorinated Biphenyls (PCB's)
EPA Methods 8081/8082

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
MB	One per preparation batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit	Determine the source of blank contamination and correct problem. If required, re-prep and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit
MS/MSD for analytes	One MS/MSD pair per analytical/preparation batch	Control limits—Attachment 2	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	Laboratory Manager / Analyst	Precisions and Accuracy	Control limits—Attachment 2
LCS or LCS/LCSD Containing Analytes Reported	One LCS or LCS/LCSD pair per analytical/preparation batch	Control limits—Attachment 2	Determine the source of the problem and correct, then re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Laboratory Manager / Analyst	Precisions and Accuracy	Control limits—Attachment 2
Surrogate Standards	Every sample, spiked sample, standard, and MB	Control limits—Attachment 2	Determine the source of problem, if related to matrix narrate or re-prep and reanalyze samples for failed surrogates in the associated preparatory batch, if enough sample material is available.	Laboratory Manager / Analyst	Accuracy	Control limits—Attachment 2

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Matrix: Soil

Organochlorine Pesticides (Pest) and Polychlorinated Biphenyls (PCB's)

EPA Methods 8081/8082

QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
			If obvious chromatographic interference with surrogate is present, then reanalysis may not be necessary.			
Confirmation of positive results (second column or second detector)	Positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis If results between primary and second column RPD ≤ 40% (report result from primary column)	If results between primary and second column RPD > 40%, then review the chromatogram, evaluate possible matrix interference on either column, and if no interference, narrate and report the lesser value with a qualifier.	Laboratory Manager / Analyst	Accuracy	Analyte concentration % difference between columns ≤ 40%

SAP WS #28.9: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 7471

Matrix: Soil
Mercury
EPA Method 7471

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > LOD	Determine source of blank contamination and correct problem. Re-prep and reanalyze calibration blank. Samples following the last acceptable calibration blank must be reanalyzed	Laboratory Manager / Analyst	Representativeness	No target analytes ≥ LOD
MB	One per digestion batch	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit	Determine source of blank contamination and correct problem. If required, re-prep and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness	No analytes detected > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit
LCS or LCS/LCSD pair if there is not enough sample for MS/MSD	One LCS or LCS/LCSD pair per each preparation batch	% Recovery 80%-124% RPD ≤ 20%	Determine the source of the problem and correct, then re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed analytes.	Laboratory Manager / Analyst	Precision / Accuracy	% Recovery 80%-124% RPD ≤ 20%
MS/MSD for analytes	One per preparatory batch per matrix	% Recovery 80%-124% RPD ≤ 20%	Determine the source of problem, if related to matrix, narrate Or re-prep and reanalyze samples for failed MS/MSD in the associated preparatory batch.	Laboratory Manager / Analyst	Precision / Accuracy	% Recovery 80%-124% RPD ≤ 20%

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Matrix: Soil

Mercury

EPA Method 7471

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Dilution test	One per preparatory batch	Five-fold dilution must agree within ±10% of the original measurement.	Evaluate for matrix interference. Perform post-digestion spike.	Laboratory Manager / Analyst	Accuracy	10% Difference, only applicable with sample Concentrations >50 × LOQ

SAP WS #28.10: LABORATORY QUALITY CONTROL SAMPLES TABLE – Method 8260

Matrix: Soil
Volatile Organic Compounds
EPA Method 8260

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
MB	One per analytical batch	No target analytes ≥ ½ LOQ For common laboratory contaminants, no analytes detected > LOQ	Determine the source of contamination and correct the problem. If required, re-prep and reanalyze MB and samples processed with the contaminated blank.	Laboratory Manager / Analyst	Representativeness, bias contamination	No target analytes ≥ ½ LOQ. For common laboratory contaminants, no analytes detected > LOQ
MS/MSD	One MS/MSD per analytical/ preparation batch	Control limits — Attachment 2	Determine if MS/MSD failure due to matrix interference. Examine the project-specific DQOs; Contact the client as to additional measures to be taken.	Laboratory Manager / Analyst	Precision / Accuracy	Control Limits (Attachment 2)
LCS or LCS/LCD	One per preparatory batch	Control limits — Attachment 2	Determine the source of LCS failure, correct the problem, then re-prep and reanalyze the LCS and samples in the associated preparatory batch for failed analytes, if enough sample material is available.	Laboratory Manager / Analyst	Precision / Accuracy	Control Limits (Attachment 2)
Surrogate standards	Every sample, spiked sample, standard, and MB	Control limits — Attachment 2	For QC and field samples, correct problem then re-prep and reanalyze failed samples for failed surrogates in the associated preparatory batch, if enough sample material is available. If obvious chromatographic interference with surrogate is present, then reanalysis may not be necessary.	Laboratory Manager / Analyst	Accuracy	Control Limits (Attachment 2)

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SAP WS #28.11: LABORATORY QUALITY CONTROL SAMPLES TABLE – CARB 435

Matrix: Soil

Asbestos

SOP/Asbestos—California Air Resources Board 435 (EM-AS-S-1265)

QC Check	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for CA	Data Quality Indicator	Measurement Performance Criteria
Blank	One MB daily	Result < Reporting Limit	Narrate/recount/reanalyze batch	Laboratory Manager / Analyst	Bias — contamination	Result < RL
Sample Duplicate	Asbestos — 1 per 50 samples	RPD < 50%	Recount batch	Laboratory Manager / Analyst	Precision	RPD < 50%
Confirmation—asbestos	10 percent of the analyzed confirmation of the quantitation result by a second analyst	Confirmation of initial results	Recount/reanalyze batch	Laboratory Manager / Analyst	Accuracy / precision	Confirmation of initial results

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SAP WS #29: PROJECT DOCUMENTS AND RECORDS TABLE

Document	Where Maintained
Final WP and SAP	EIP: onsite and/or Project Sharepoint NAVFAC SW Environmental Restoration Program Record File for CERCLA sites
Field notes/logbook	EIP: onsite and/or Project Sharepoint
Chain of Custody Forms	EIP: onsite and/or Project Sharepoint NAVFAC SW Environmental Restoration Program Record File for CERCLA sites (at end of project – included as part of laboratory's raw data package)
Laboratory raw data package	EIP: Project Sharepoint Final RACR NAVFAC SW Environmental Restoration Program Record File for CERCLA sites (at end of project)
Laboratory report Corrective action report Sample preparation Run logs CLP-equivalent (Stage 4) analytical laboratory reports, including raw data	EIP: Project Sharepoint Analytical laboratory NAVFAC SW Environmental Restoration Program Record File for CERCLA sites (at end of project)
Data validation reports	EIP: Project Sharepoint Final RACR NAVFAC SW Environmental Restoration Program Record File for CERCLA sites (at end of project) Validated electronic data will be loaded into Naval Installation Restoration Information Solution, the Navy's centralized database (at end of project)

Notes:

Project Sharepoint: EIP utilizes the cloud-based Microsoft Sharepoint file system. Electronic copies of all project documents will be stored in this location.

Active project files will be maintained by EIP until the project is completed. Following project completion, if hardcopy files are produced, they will be stored for a minimum of 10 years EIP project management

Documents submitted to the NAVFAC SW Environmental Restoration Program Record File are located at:

Commanding Officer
Naval Facilities Engineering Command, Southwest
1220 Pacific Highway (Naval Base San Diego Bldg. 3519)
San Diego, CA 92132

Following response complete at the facility, hardcopy deliverables will be archived by the Navy at a Federal Records Center (http://www.archives.gov/frc/locations.html) where they are maintained for 50 years.

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SAP WS #30: ANALYTICAL SERVICES TABLE

Matrix	Analytical Group	Sample Locations/ ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory/ Organization	Backup Laboratory
Soil (Radiological)	Gamma Isotopes Strontium (^{90}Sr) Alpha isotopes (additional isotopes as necessary – per decisions in WS#10) Tritium (^{3}H)	Applicable radiological samples shown in WS #18	901.1 905.0 DOE Method A-01-R 906.0	30 calendar days	Eurofins Test America, St. Louis Laboratory [1] 13715 Rider Trail North Earth City, Missouri 63045 314-298-8566	None[2]
	Gamma Isotopes Strontium (^{90}Sr)	Import Fill • WS #17-2	901 905			
Soil	PCBs TPH Metals PAHs Pest SVOC VOCs	Import Fill • WS #17-2	8082A 8015B 6020/7471A 8270C-SIM 8081A 8270C 8260B	21 days after sample delivery	Eurofins Test America Denver 4955 Yarrow St Arvada, Colorado 80002 303-736-0100	None
Soil	Asbestos	Import Fill • WS #17-2	CARB 435	21 days after sample delivery	NVL Laboratory 4708 Aurora Avenue North, Seattle, Washington 98103 206-547-0100	None

Note: [1]DoD Environmental Laboratory Accreditation Program accreditation certificates are provided in Attachment 2. Status of laboratory certifications/accreditations will be verified prior to fieldwork and before samples are delivered to the laboratory. Updates to laboratory accreditation to ensure the laboratory is qualified to perform the analysis will be made prior to sample testing.

[2]A backup laboratory has not been identified. If circumstances render the subcontracted laboratory unable to perform the analytical services, then another laboratory will be determined at that time.

SAP WS #31: PLANNED PROJECT ASSESSMENTS TABLE

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment	Person(s) Responsible for Responding to Assessment Findings	Person(s) Responsible for Identifying and Implementing Corrective Actions	Person(s) Responsible for Monitoring Effectiveness of Corrective Action
Initial inspection / preparatory meeting	Prior to the start of sampling activities	I	EIP	EIP personnel (designated by Project PM, PgQCM, or PQCM)	EIP Project Chemist or Sample Technician	EIP Project Chemist or Sample Technician	EIP Program Chemist or Project QC Manager
Field Sampling Technical Systems Audit	At least one field Technical Systems Audit at the start of field activities	I	EIP and/or Navy QAO	EIP personnel designated by Project PM, PgQCM, or PQCM	EIP Project Chemist or Program Chemist	EIP Project Chemist or Program Chemist	EIP Project Chemist or Program Chemist
Laboratory data review/validation for compliance to SAP requirements	Ongoing throughout the project as data is received from the laboratory	I	EIP - Designated Data Validation Sub-contractor	Data Validation PM	EIP Project or Program Chemist	Laboratory PM via EIP chemist or EIP Chemist	EIP Project or Program Chemist
Field documentation and data review	At least once at the beginning of sampling activities and then as needed as the project progresses or if requested by the Project Technical Lead or PM	I	EIP	Project QC Manager or Project Chemist	EIP QC Manager, or Project Chemist, or Field Sampling Technician,	EIP Technical Manager, Field Sampling Technician, or Project Chemist	EIP Program Chemist or Project QC Manager
QC	Weekly	I	EIP	Project QC Manager	Project Staff	Program or Project QC Manager	Project QC Manager

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SAP WS #32: ASSESSMENT FINDINGS AND CORRECTIVE ACTION RESPONSES

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response	Timeframe for Response
Initial inspection/ preparatory meeting	ORR Checklist	EIP PM	As soon as possible, within same day of finding	ORR Checklist with outstanding actions completed or addressed prior to project work.	EIP PM	1 business day
Field Sampling Technical System Audit	Written audit report	EIP PQCM	48 hours after audit	Email or letter	Field Technician, ECC Project Chemist, ECC Program Chemist, EIP PM	24 hours after notification
Laboratory Data Review Findings	Data validation reports	EIP Project Chemist	48 hours after data review of data has been completed	Email or letter	Primary: EIP Project Chemist, EIP Program Chemist, and or EIP Project Health Physicist Secondary (if required): Laboratory QA Manager or Laboratory PM.	3 days after notification

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SAP WS #33: QUALITY ASSURANCE MANAGEMENT REPORTS TABLE

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
Field Sampling, Audit Report Initial and Follow-Up Inspections	At least once at the beginning of sampling activities and then as needed as the project progresses	Within 24 hours of Field Sampling Audit	EIP Project QC Manager or EIP Project Chemist	EIP PM
DQA • Provides an overview of sampling, decontamination, and data storage procedures • Identifies QC samples and summarizes associated analytical results • Summarizes the findings of the analytical data validation process Provides an evaluation of data quality in accordance with the data quality indicator (DQIs) as defined in the SAP	Once for all data per parcel	Approximately 60 days after field investigation is complete	EIP Program Chemist; EIP RSO; EIP Project Chemist; or Personnel designated by EIP PM	Navy LRPM/BLTL EIP PM
Final Project Report	After completion of fieldwork	Project document delivery schedule is provided in the WP	EIP Project Chemist, EIP Program Chemist, or personnel designated by the EIP PM.	EIP PM
Data Validation Report	After data packages are received from laboratory	Within two weeks of data package receipt	Independent third-party data validation company	EIP Project Chemist or EIP Program Chemist
Final Project Report and Data Quality Assessment Report	After completion of fieldwork	Project document delivery schedule is provided in the WP	EIP Project Manager	Navy RPM and regulatory agencies (WS #3)

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SAP WS #34, WS #35, WS #36: DATA VERIFICATION AND VALIDATION (STEPS I AND IIA/IIB) PROCESS TABLE

Data Review Input	Description	Responsible for Verification	Step I/IIa/IIb [1]	Internal/External
Chain of Custody Forms	Chain of Custody Forms will be reviewed internally upon their completion and verified against the packed sample coolers they represent. The shipper's signature on the Chain of Custody Form should be initialed by the reviewer, a copy of the Chain of Custody Form retained in the project file, and the original inside the cooler for shipment.	Field sampling team leader (EIP) and/or Project Chemist	Step I	Internal
Sample receipt	The sample cooler will be checked for compliance with preservative, temperature and packaging requirements. Sample containers will be reviewed against the chain-of-custody for agreement. Sample receipt will be documented by the laboratory on a login sheet and sample information will be entered into the Laboratory Information Management System.	Laboratory sample receiving and Laboratory PM	Step I	External
Field notes / logbook	Field notes will be reviewed internally and placed in the project file upon project completion.	EIP Project Chemist and/or Project QC Manager	Step I	Internal
Audit reports	Upon report completion, a copy of audit reports will be placed in the project file. If corrective actions are required, then a copy of the documented corrective action taken will be attached to the appropriate audit report in the project file. At the beginning of each week, and at the completion of the site work, project file audit reports will be reviewed internally to ensure that appropriate corrective actions have been taken and that corrective action reports are attached. If corrective actions have not been taken, then the PM will be notified to ensure action is taken.	EIP Project QC Manager, Construction Manager, or EIP PM	Step I	Internal
Laboratory data packages	Laboratory data packages will be verified internally by the laboratory performing the work for completeness and technical accuracy prior to submittal. Received data packages will be verified by the EIP Chemist and a third-party reviewer according to the data validation procedures specified in this SAP.	Laboratory PM, EIP Chemist, Data Validation PM	Step I	Internal/External
EDD	EDDs will be verified internally by the subcontract laboratory for completeness and technical accuracy prior to submittal to EIP. EDDs will be verified by the validation contractor against the hardcopy laboratory reports (10% checked). Discrepancies will resolved by the laboratory.	Laboratory, EIP Chemist, and a third-party data validation company	Step I	Internal/External
Sampling Methods and Procedures	Ensure that the required sampling methods were used to collect project samples, field changes or deviations are noted in the field logbook. Review field sample collection logbooks for compliance with the approved SAP.	EIP Project Chemist and Project QC Manager	Step IIa	Internal
Holding times	Ensure the samples were analyzed within the EPA holding times. If holding times were not met, then verify that deviations were documented and proper notifications were made.	Laboratory PM	Step IIa	External

Data Review Input	Description	Responsible for Verification	Step I/IIa/IIb [1]	Internal/External
Analytes and Project DLC are met	Ensure that the required list of analytes and that project-specific DLC specified in this SAP are met and reported per project requirements.	Laboratory PM, EIP Chemist, and/or Third-Party Data validation company	Step IIa	Internal/External
Electronic Sample Data Packages	Review data package for compliance with EPA method requirements (1996, 2008) and DoD QSM 5.3 (2019) requirements, and approved SAP.	Third-Party Data validation company	Step IIa	External
Documentation of EPA Method QC sample results; 90% Stage 2B	Determine if EPA Method required QC samples were analyzed and met required control limits per SAP and DoD QSM 5.3 (2019) requirements when applicable.	Third-Party Data validation company	Step IIa	External
10% Stage 3/4 Raw Data Review	Recalculation of laboratory raw data to confirm laboratory calculation of final results.	Third-Party Data validation company	Step IIa	External
Documentation of SAP QC sample results	Determine if SAP required QC samples were collected and met required control limits per SAP requirements.	Third-Party Data validation company	Step IIb	External
Sampling Plan	Determine whether the SAP was executed as specified (number, location, type of field samples collected).	EIP Project QC Manager or Project Chemist	Step IIb	Internal
Field duplicate precision	Compare results for field duplicates with criteria established in this SAP.	Third-Party Data validation company	Step IIb	External
Analytical data for radiological parameters in samples	Data validation in accordance with DoD *General Data Validation Guidelines* (DoD, 2019a), DoD *Data Validation Guidelines, Module 1* (DoD, 2020), National Functional Guidelines (2020a and 2020b), Multi Agency Radiological Laboratory Analytical Protocols Manual (MARLAP) (MARLAP; EPA et al., 2004), and QC criteria in project SAP, • MARSSIM (EPA 402-R-97-016, 2000). and DoD QSM 5.3 (2019)	Third-Party Data validation company	Step IIb	External

Notes: [1] IIa = compliance with methods, procedures, and contracts (see Uniform Federal Policy for Quality Assurance Project Plans, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs [EPA, 2005] Table 10, page 117)

IIb = comparison with measurement performance criteria in the SAP (see Uniform Federal Policy for Quality Assurance Project Plans, Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs [EPA, 2005] Table 11, page 118)

Data Validation will be presented in project specific reports per sample data / laboratory data package

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Validation of Laboratory Data

Data validation is a systematic, independent process of reviewing a body of data to determine the analytical limitations of that data based on specific QC criteria. A third-party data validation company will validate definitive-level project laboratory data for primary site chemicals of concern at 90 percent Stage 2B and full validation at 10 percent at Stage 3/4.

The EIP Project Chemist will specify the sample and/or sample groups using a random selection process for the 10 percent Stage 3/4 reporting and validation. Critical samples, if identified, or other samples determined to be of decision-making significance (as determined by the Project Chemist or Project Technical Lead), will be included in the 10 percent.

The data review, validation and qualification will be in compliance with the following guidance:

- DoD QSM 5.3 (2019)
- SAP
- *General Data Validation Guidelines* (DoD, 2019)
- *Data Validation Guidelines, Module 1* (DoD, 2020)

Additional validation guidance will be obtained from the following:

- MARLAP (EPA et al., 2004)
- *USEPA Contract Laboratory Program National Functional Guidelines for Organic Superfund Data Review* (EPA, 2020a)
- *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* (EPA, 2020b)

Data will be flagged with the following data qualifiers:

- **J** qualifier denotes the analyte was positively identified, but the associated numerical value is estimated.
- **U** qualifier denotes the analyte was analyzed for, but not detected at or below the limit of detection.
- **X** qualifier denotes the sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended
- **N** qualifier denotes the analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- **NJ** qualifier denotes the analyte has been "tentatively identified" or "presumptively" as present, and the associated numerical value is the estimated concentration in the sample
- **UJ** qualifier denotes the analyte was analyzed for, but not detected and there is uncertainty associated with the reporting limit.

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The Project Chemist or reviewer's professional judgment will be used to evaluate data quality when called for in the guidance documents above. Professional judgment will also be used where no clear policy exists, or when there is conflicting guidance on how data should be qualified.

Stage 2B and Stage 3 Data Review and Validation Checklist

Data quality is assessed by comparing the parameters listed below to the appropriate criteria (or limits) as specified in the project SAP, DoD QSM 5.3 (2019), or by EPA method-specific requirements. If calculations for quantitation are verified, then it is done on a limited basis requires raw data (Stage 3/4) in addition to the standard data forms normally present in a data package.

Data validation reports include outliers for QC criteria evaluated and data qualifiers added to sample results for each outlier. Data review/validation may include the following QC elements shown in the example validation checklist shown below (depending on the analysis being reviewed):

Radiological Analyses

Quality Control Check	Evaluation	Data Qualification	Samples Affected
Holding Time	N/A	None required	
Sample Preservation	N/A	None required	
Temperature	N/A	None required	
ICAL (See Worksheet #24 for criteria)			
Energy	Energy difference outside criteria	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Associated analytes in samples in analytical batch
Efficiency	Efficiency difference outside criteria	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
FWHM peak resolution	FWHM peak resolution outside criteria	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
ICV	Observed peaks in ICV greater than 10% of ICAL value	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Associated analytes in samples in analytical batch
CCV (Daily Check)	Energy, efficiency, or FWHM outside criteria	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Associated analytes in samples in analytical batch
BSC	Background count rate of entire spectrum > 3σ of the average	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Associated samples in analytical batch
Instrument Contamination Check	Background count rate of entire spectrum > 3σ of the average	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Associated samples in analytical batch

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Radiological Analyses

Quality Control Check	Evaluation	Data Qualification	Samples Affected
LCS	%R >UCL	Sample > DLC; qualify as estimated (J) Sample < DLC; None required	Associated analytes in samples in preparation batch or analytical batch
	%R <LCL but ≥ 30%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
	%R < 30%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as unusable (X)	
Method Blank	Blank < DLC	None required	Associated analytes in samples in preparation batch or analytical batch
	Blank > DLC	Sample < DLC; None required Sample > DLC by < 10x blank; qualify as estimated (J) Sample > 10x blank; None required	
Tracer Recovery (alpha spectroscopy only) Carrier Recovery (GFPC only)	%R > UCL	Sample > DLC; qualify as estimated (J) Sample < DLC; None required	Associated analytes in affected samples
	%R < LCL but ≥ 10%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
	%R < 10%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as unusable (X)	
Laboratory Control Sample Duplicates	Concentration of reported analytes are > 5x the DLC in either sample and RPD ≥ 25% and/or RER ≥ 1	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Analytes in parent sample
	Concentration of reported analytes are < 5x the DLC in either sample and absolute difference > 3x DLC	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
Matrix Spike (if performed) if activity of the sample > 5 times the spiking level.	%R >UCL	Sample > DLC; qualify as estimated (J) Sample < DLC; None required	Associated analytes in samples in preparation batch or analytical batch
	%R <LCL but ≥ 30%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	
	%R < 30%	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as unusable (X)	

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Radiological Analyses

Quality Control Check	Evaluation	Data Qualification	Samples Affected
Field Duplicates	Concentration of reported analytes are > 5x the DLC in either sample and RPD ≥ 25% and/or RER ≥ 1	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	Analytes in parent sample and field duplicate
	Concentration of reported analytes are < 5x the DLC in either sample and absolute difference > 3x DLC	Sample > DLC; qualify as estimated (J) Sample < DLC; qualify as estimated (UJ)	

Notes: Backfill chemical analysis validation will the use checklist as presented in validation guidance documents DoD General Validation (2018) and National Functional Guidelines for Organic and Inorganic Superfund Data Review (2020a and b)

QA/QC criteria are included in WS #12, WS #24, and WS #28 and will be used for validation criteria.

%R = percent recovery
LCL = lower control limit
UCL = upper control limit

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SAP WS #37: USABILITY ASSESSMENT

The DQO for the project include the following goals:

- To evaluate and document the validity of the obtained radiological soil data to support decisions
- To confirm prior investigation results
- To compare radiological data to RGs
- To recommend remediation if necessary
- To compare radiological data to applicable RG or natural background values (whichever is higher)

Assessment of sampling and survey data consists of four separate and identifiable phases: data reduction, data verification, data validation, and data quality assessment (DQA). These processes will be performed in accordance with MARLAP (EPA et. al, 2004) and other applicable guidance. Data reduction involves data transformation processes such as converting raw data into reportable quantities and units, using significant figures, and calculating measurement uncertainties. Verification and validation pertain to evaluation of survey and analytical data and are considered as two separate processes.

Data verification compares the survey and sampling data collection against the requirements of the project-specific WP and SOPs. For example, the actual survey locations, scan speed, number and location of systematic static survey measurements, and the number and location of swipe samples will be compared with the planned survey activities. A verification report may be prepared depending on the size and complexity of the survey. The verification report identifies those requirements that were not met (called exceptions). Task-specific verification checklists will be developed prior to field mobilization to ensure that requirements identified in the work planning documents are met. Data verification also involves reviewing data that was transcribed or transferred into the electronic data management systems. The data verification will be performed by the Project Radiation Safety Officer and other senior staff with access to the original data, SOPs, and the WP.

At HPNS, the verification process will include the following:

- Appropriate selection of the survey instruments
- Appropriate survey methods for the ROCs
- Evaluation of data completeness
- Verification of instrument/detector calibration
- Daily response checks of the instrument/detector

- Assessment of survey method specifications, including scan speed, distance from the detector to surveyed surface, survey path, time that counts are collected, and adherence to operator response requirements, such as response to measurements exceeding the investigation level and documentation of adverse conditions:

  - Retrospective calculation of DLCs
  - Adjustments of background count rate settings
  - Checks on instrument system performance
  - Swipes collected as required: labeling, analyses, and documentation
  - Recorded measurement and sample locations per project requirement

Validation is a systematic check on the set of survey or analytical data being used to meet the project requirements and is performed to addresses the usability of the data. The validation process begins with a

review of the survey or analytical data package to identify its areas of strength and weakness. The validation process should determine the impact of not meeting the requirements of the Parcel E WP and SOPs. Validation then evaluates the data to determine the absence of a required survey measurement (i.e., laboratory results), the uncertainty of the survey process, and the technical reliability and the degree of confidence in the reported survey data are considered. The validator will note if there is data that does not meet the performance criteria (WS #28). The products of the validation process are validated data, which include the data validation reports which identified the data that has met the review criteria and those that did not meet the review criteria. Unique data issues are also summarized for use in the evaluation process.

37.1 Data Quality Assessment Report

The DQA is the last phase of the data collection process and consists of a scientific and statistical evaluation of project-wide knowledge to assess data usability. DQA considers sampling, analytical, and data handling details, external QA assessments, and other historical project data to determine the usability of data for decision-making. To assess and document overall data quality and usability, the data quality assessor integrates the data validation report, field information, assessment reports, and historical project data, and compares the findings to the DQOs objectives defined in the WP and this SAP. The DQA process uses the combined findings of these multi-disciplinary assessments to determine data usability for the intended decisions, and to generate a DQA report documenting that usability and the causes of deficiencies.

The DQA process varies depending on the survey objectives and the level and depth of the verification. The process will evaluate and document the usability of the data by considering the project data quality indicators (DQIs), which are precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS). The DQA process will determine whether the data will be suitable for the intended needs of the project. Every data type (e.g., sampling, field screening data, and laboratory analytical data) will be relevant to the usability assessment. Data usability will include the entry of analytical data validation flags, applied by the third-party analytical data validation subcontractor, to the project data, as well as an overall assessment of the analytical data and field QC samples.

The assessment will consider the relationship of each type of data to the entire data set, and the adequacy of the data to fulfill the project DQOs. The data will be assessed for correctness, completeness, and compliance to method-specific or project-specific QA/QC requirements, including the results of the independent analytical data validation process and contractual requirements. Analytical data validation will evaluate the data based on the PARCCS criteria defined in this SAP and other method-specific performance requirements. The overall assessment process will also evaluate data usability based on the intended use of the data. The intent of the DQA process will be to evaluate the data against the PARCCS levels and establish the usability of the final results with respect to the project DQOs. Upon completion of analytical data validation, each data point will be assessed as non-qualified, qualified as estimated ("J" or "UJ" qualified), or qualified as rejected ("R" qualified) based upon the acceptance criteria, and analytical data validation flags will be added to the project data. These parameters will be based on the analytical data quality and will encompass the DQIs established in this SAP. Qualification will be given according to each sample's delivery group and will be based on the SAP and applicable laboratory and data validation procedures. Both analytical and contractual compliance and completeness levels will be assessed for each analytical parameter. Finally, the overall usefulness of the data will be established as related to the project DQOs.

The Project Team will review and evaluate the DQA/data usability assessment, which will be provided in the Final Project Report.

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The data will be evaluated for overall PARCCS criteria for each matrix, analytical group, and concentration level, and data use limitations will be discussed in the DQA/data usability assessment reports for data that do not meet the project DQOs or DQIs. The DQA/data usability assessment reports will include a detailed discussion of the data usability evaluations with sufficient information to support the data usability conclusions, such as the following:

- Review of data reduction, verification, and validation
- Assessment of trends and biases equilibrium of radionuclide decay chains
- Analysis of environmental radioactivity
- Variations of natural radionuclides
- Satisfaction of quality objectives
- Overall defensibility and usability
- Appropriate analysis to support usability

The level of data verification, validation, and DQA performed on radiological samples is defined in WS #34 through WS #36. Copies of surveys, sampling, and analytical data (and their supporting data) will be protected and maintained in project record files.

37.2 Data Quality Indicators

The quality of analytical data is measured using data quality criteria. Data quality criteria include the following: precision, accuracy, representativeness, comparability, completeness, and sensitivity. The following discussion of data quality criteria is for definitive-level data, though the same criteria (but not necessarily the same procedures, units, or values) may also be applied to screening level analytical data.

37.2.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. The following equation illustrates the method for calculating RPD to assess a method's precision:

$$\text{Precision as RPD} = \frac{2\times(\text{Result-Duplicate Result})\times 100\%}{\text{Result + Duplicate Result}}$$

The laboratory(s) uses sample duplicates or tracer assess the precision of analytical procedures. For radiochemistry analysis MS/MSD samples are not performed if tracer compounds are used.

The sample duplicates or tracers and are prepared and analyzed with each batch of samples instead of MS/MSD. For inorganic analyses, analytical precision is usually calculated based on the sample and sample duplicate results.

The analytical laboratory(s) will use DoD QSM 5.3 (2019) control limits if available; otherwise, the laboratory(s) will have statistically based acceptability limits for RPDs established for each method of analysis and sample matrix. The laboratory(s) will review the QC samples to ensure that internal QC data lies within the limits of acceptability. Suspect trends will be investigated and corrective actions will be implemented, if warranted.

37.2.2 Accuracy

Accuracy measures the bias of an analytical system by comparing the difference of a measurement with a reference value. The percent recovery of an analyte, which has been added to the environmental samples

at a known concentration before extraction and analysis, provides a quantitation tool for analytical accuracy. The spiking solutions used for accuracy determinations are not used for instrument calibrations. The following equation illustrates how accuracy is evaluated:

$$\text{Accuracy as percent recovery} = \frac{\text{Spiked Sample Result-Sample Result} \times 100\%}{\text{Spiked Sample True Value}}$$

Percent recoveries for MS/MSD (optional), and LCS that are analyzed for every batch of up to 20 samples serve as a measure of analytical accuracy. Surrogate standards are added to chemical samples, blanks, MSs, MSDs, and LCSs analyzed for organic contaminants to evaluate the method's accuracy and to help determine matrix interferences. For radiological samples, carriers or tracers are added to evaluate the methods accuracy and help determine matrix interference.

The laboratory(s) may use statistically based or in-house control limits are developed for each method of organic and radiological analysis and sample matrix.

The laboratory(s) will review the QC samples recoveries for each analysis to ensure that internal QC data lie within the limits of acceptability. The laboratory(s) will investigate suspect trends and take appropriate corrective actions.

37.2.3 Representativeness

Unlike precision and accuracy, which can be expressed in quantitative terms, representativeness is a qualitative parameter. Representativeness is the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. It is a qualitative parameter that depends on proper design of the sampling program.

Field personnel will be responsible for ensuring that samples are representative of field conditions by collecting and handling samples according to this SAP, the chemical data quality management plan, and field SOPs. Errors in sample collection, packaging, preservation, or chain-of-custody procedures may result in samples being judged non-representative and may form a basis for rejecting the data.

Data generated by the laboratory(s) must be representative of the laboratory(s) database of accuracy and precision measurements for analytes in different matrices. Laboratory(s) procedures for sample preparation will ensure that aliquots used for analysis are representative of the whole sample.

37.2.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another, whether it was generated by a single laboratory(s) or during inter-laboratory(s) studies. The use of standardized field and analytical procedures ensures comparability of analytical data.

Sample collection and handling procedures will adhere to EPA-approved protocols. Laboratory(s) procedures will follow standard analytical protocols, use standard units, standardized report formats, follow the calculations as referenced in approved analytical methods, and use a standard statistical approach for QC measurements.

37.2.5 Completeness

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Completeness is a measure of whether the data necessary to meet the project have been collected. For the data to be considered complete, they must meet acceptance criteria including accuracy and precision and other criteria specified for an analytical method. The data will be reviewed and/or validated to keep rejected (invalid data) from being used in project decisions. Completeness will be calculated per analyte per method. A full sample will not be rejected if one or more analytes fails to meet acceptance criteria. Completeness is evaluated using the following equation:

$$\text{Completeness} = \frac{\text{Acceptable Analytes reported and validated} \times 100\%}{\text{Total Number of Analytes Collected}}$$

The goal for completeness is 90 percent of the validated data being acceptable (i.e., not rejected) as qualified. Only data analyzed within holding times will be used for this project. If these goals are not achieved, then the sources of non-conformances will be evaluated to determine whether re-sampling and reanalysis are necessary.

Field completeness will be calculated based on the number of samples collected divided by the number of samples planned to be collected. The sampling density will meet the requirements stated in WS #17.

37.2.7 Sensitivity

Sensitivity is the capability of a method or instrument to measure target analyte responses. Sensitivity determines the minimum concentration or attribute that can be measured by a method (method detection limit), by an instrument (instrument detection limit), or by a laboratory(s) (Decision Level Concentration (DLC)). The laboratory(s) DLC will be sensitive enough to meet the project decision limits. The laboratory(s) will determine the DLC for each method, instrument, analyte, and matrix by using the procedure described by their analytical SOPS. The laboratory(s) DLC will be below the project RGs (WS #15).

The laboratory(s) DLC is a value at which there is at most a 5 percent probability of incorrectly deciding that the analyte is present. Note that for most instrumentation, the DLC is approximately half the value of the DLC. Sensitivity may be affected by sample matrix factors such as interference of non-target analytes, sample dilution or moisture content (for soil samples).

The project chemist will evaluate the DLC prior to sample analysis to determine if the laboratory(s) is able to attain the required sensitivity for the project. The DLC will be evaluated after sample analysis to determine if there were matrix effects, operator errors, or analytical process errors that interfered with the ability to compare the results to the project decision limits. Reported results will be reviewed and if the verification and validation are acceptable, then the data is usable. If anomalies in sensitivity are present, then the rationale for use or non-use of the affected samples will be discussed in the DQA report.

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Figures

Figure 1

Site Location Map

Figure 2

Parcel E – Phase 1 and 2 Trench Units, Former Building Sites, and Existing Buildings

Figures 3 thru 5

Additional Laboratory Analyses Decision Tree

Figure 3 - Radium 226

Figure 4 - Cesium 237

Figure 5 - Strontium 90

Attachment 1
Field Forms

(provided on electronic copy only)

- Standard Drilling Log (Word and PDF copies)
- Sample Chain of Custody
- Rad-Chem Field Measurement Forms

Attachment 2
Laboratory Standard Operating Procedures

(provided on electronic copy only)

Attachment 3

Field Sampling SOPs

(provided on electronic copy only)

- **ENV-601 Sample Handling and Custody Requirements**
- **ENV 603 Field Sample Documentation (Draft)**
- **ENV-610 Field Quality Control Samples**
- **ENV-801 Soil Sampling (Draft)**
- **ENV-802 Soil Characterization/Classification**
- **RP-104 Radiological Surveys (Swipe Samples / Section 10.3)**
- **RP 108 Count Rate Instruments**
- **RP 109 Dose Rate Instruments**

Figures

Figure 1

Site Location Map

Legend

- Parcel E
- Other Parcels
- Parcel F (Water) Boundary

Service Layer Credits: Sources: Esri, DeLorme, NAVTEQ, USGS, Intermap, iPC, NRCAN, Esri Japan, METI, Esri China (Hong Kong), Esri (Thailand), TomTom, 2013
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N

0 450 900 1,800 Feet

1 inch = 900 feet

Figure 1-1
HPNS and Parcel E Location

Parcel E Sampling and Analysis Plan
Former Hunters Point Naval Shipyard
San Francisco, California

Figure 2

Parcel E – Phase 1 and 2 Trench Units, Former Building Sites, and Existing Buildings

Legend

- Parcel E Boundary
- Existing Impacted Buildings
- Demolished Impacted Buildings
- Phase 1 Trench Units
- Phase 2 Trench Units

Service Layer Credits: Sources: Esri, HERE, Garmin, USGS, Intermap, INCREMENT P, NRCan, Esri Japan, METI, Esri China (Hong Kong), Esri Korea, Esri (Thailand), NGCC, (c) OpenStreetMap contributors, and the GIS User Community
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N

0 150 300 600 Feet

1 inch = 300 feet

Figure 2
Phase 1 and 2 Trench Units, and Building Sites

Parcel E Sampling and Analysis Plan
Former Hunters Point Naval Shipyard
San Francisco, California

Figures 3 thru 5

Additional Laboratory Analyses Decision Tree

Figure 3 - Radium 226

Figure 4 - Cesium 237

Figure 5 - Strontium 90

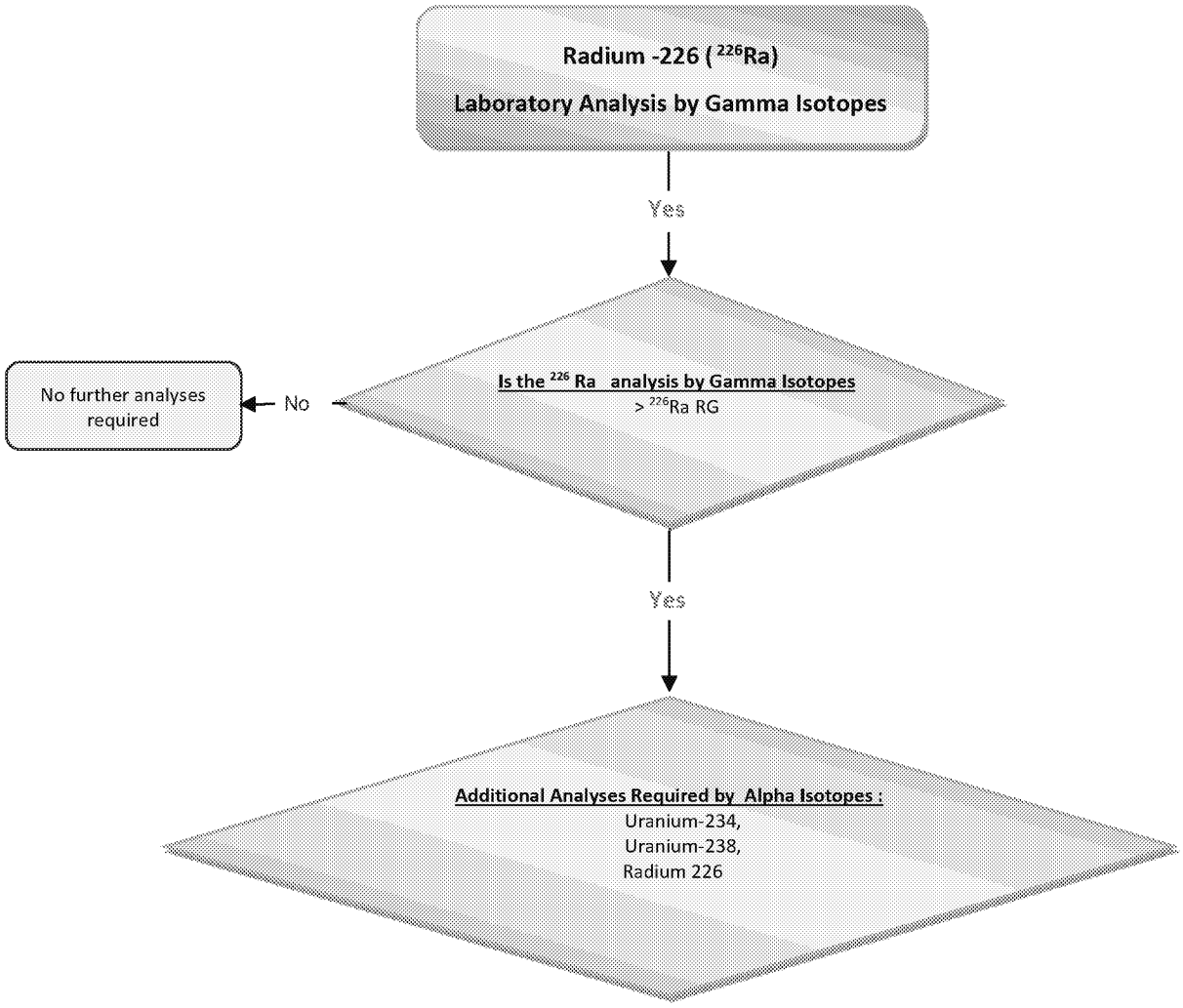
Figure 3

Hunters Point – Parcel E

Primary Analysis: Radium 226 by Gamma Isotopes

Secondary Analyses: Uranium-234 , Uranium-238, 226-Radium by Alpha Isotopes

Additional Laboratory Analyses Decision Tree



If the concentrations of the radionuclides in the uranium natural decay series are consistent with the assumption of secular equilibrium, then the Radium 226 concentration is NORM and site conditions comply with the ROD RAO. .

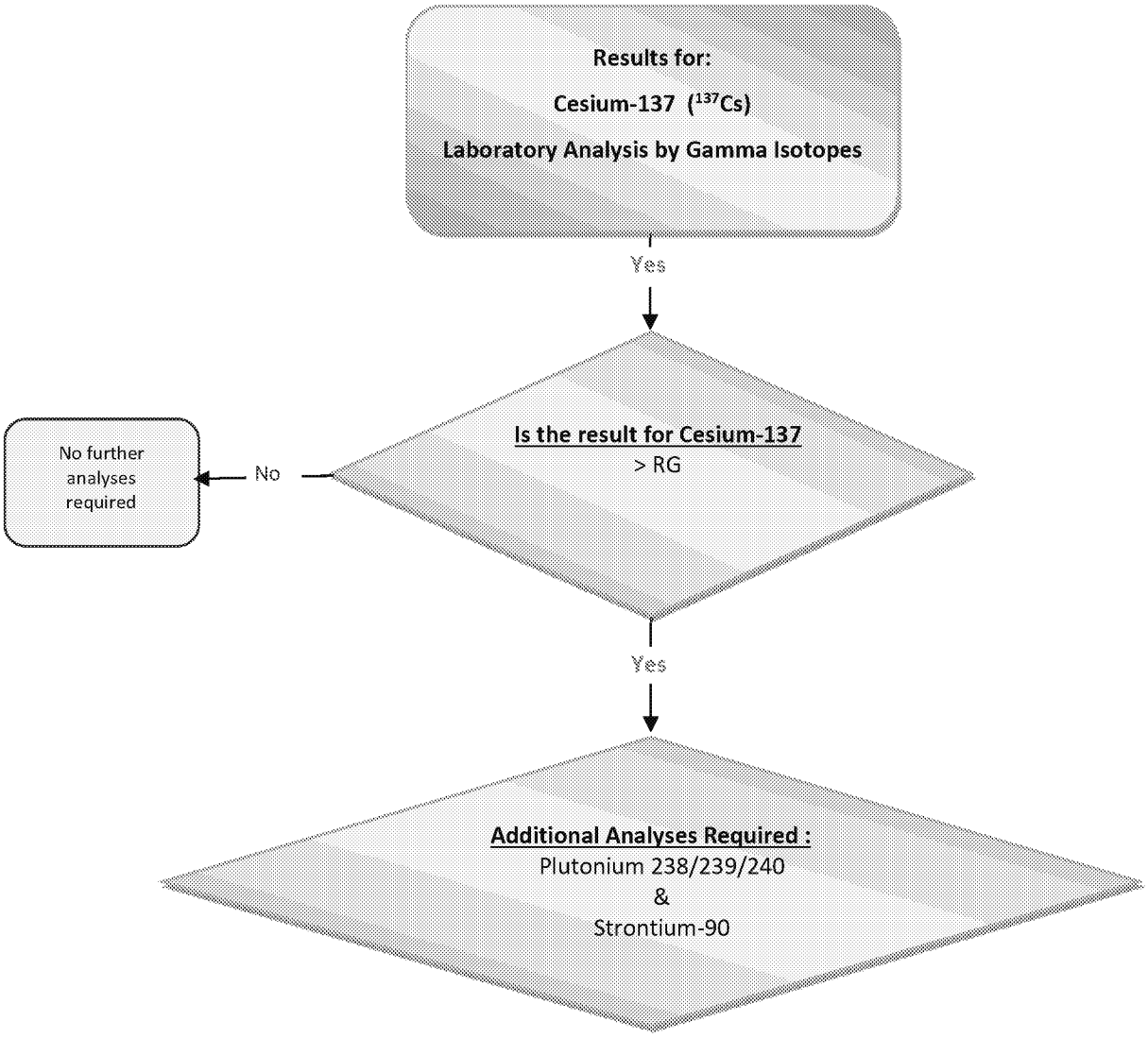
Figure 4

Hunters Point – Parcel E

Primary Analysis: Cesium-137

Secondary Analyses: Plutonium-238/239/240

Additional Laboratory Analyses Decision Tree



If the concentrations of the radionuclides in the uranium natural decay series are consistent with the assumption of secular equilibrium, then the Radium 226 concentration is NORM and site conditions comply with the ROD RAO. .

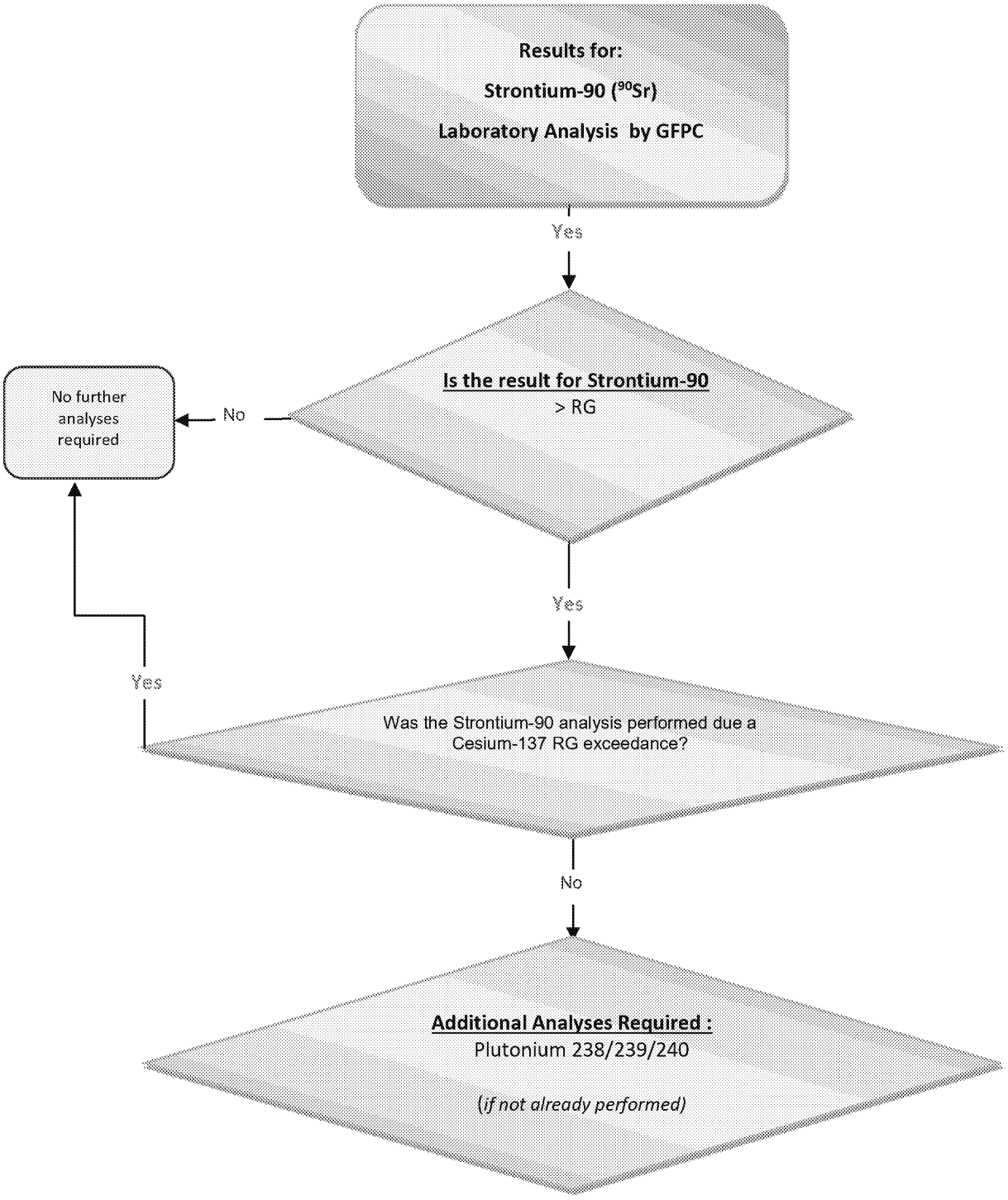
Figure 5

Hunters Point – Parcel E

Primary Analysis: Strontium-90

Secondary Analyses: Plutonium-238/239/240

Additional Laboratory Analyses Decision Tree



If the concentrations of the radionuclides in the uranium natural decay series are consistent with the assumption of secular equilibrium, then the Radium 226 concentration is NORM and site conditions comply with the ROD RAO. .

Attachment 1
Field Forms

(provided on electronic copy only)

- Standard Drilling Log (Word and PDF copies)
- Sample Chain of Custody
- Rad-Chem Field Measurement Forms

DRILLING LOG

BORE HOLE _____
INSTALLATION _____
PROJECT NUMBER _____ DATE _____
LOCATION _____ GEOLOGIST _____
_____ DRILLERS _____
_____ _____
_____ DRILL RIG _____
_____ _____
NOTES _____

DEPTH (feet)	DESCRIPTION	REMARKS
0		
5		
10		
15		
20		

TestAmerica St. Louis
13715 Rider Trail North

Earth City, MO 63045-1205
phone 314.298.8566 fax 314.298.8757

Chain of Custody Record

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Regulatory Program: ☐ DW ☐ NPDES ☐ RCRA ☐ Other:

TestAmerica Laboratories, Inc.

Client Contact	Project Manager:	Site Contact:		COC No:
	Tel/Fax:	Lab Contact:	Carrier:	___1___ of ___1___ COCs
	Analysis Turnaround Time			Sampler:
	☐ CALENDAR DAYS ☐ WORKING DAYS			For Lab Use Only:
Ph	TAT if different from Below Please turn around per our conversation			Walk-in Client:
fax	☐ 2 weeks			Lab Sampling:
Project Name:	☐ 1 week			
Site:	☐ 2 days			Job / SDG No.:
P O	☐ 1 day			

Sample Identification	Sample Date	Sample Time	Sample Type (C=Comp, G=Grab)	Matrix	# of Cont.	Filtered Sample (Y / N)	Perform MS / MSD (Y / N)	Gamma Spec - See Attached Note																Sample Specific Notes:

Preservation Used: 1= Ice, 2= HCl; 3= H2SO4; 4=HNO3; 5=NaOH; 6= Other _____

Possible Hazard Identification:
Are any samples from a listed EPA Hazardous Waste? Please List any EPA Waste Codes for the sample in the Comments Section if the lab is to dispose of the sample.

☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☐ Unknown

Sample Disposal (A fee may be assessed if samples are retained longer than 1 month)

☐ Return to Client ☐ Disposal by Lab ☐ Archive for_____ Months

Special Instructions/QC Requirements & Comments: Please review attached instructions.

Custody Seals Intact: ☐ Yes ☐ No | Custody Seal No.: | Cooler Temp. (°C): Obs'd:_____ Corr'd:_____ Therm ID No.:_____

Relinquished by:	Company: Perma-fix	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received by:	Company:	Date/Time:
Relinquished by:	Company:	Date/Time:	Received in Laboratory by:	Company:	Date/Time:

Form No. CA-C-WI-002, Rev. 4.9, dated 2/2/2016

Attachment 2
Laboratory Standard Operating Procedures

(provided on electronic copy only)

CALIFORNIA
Water Boards
STATE WATER RESOURCES CONTROL BOARD
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

Eurofins TestAmerica St. Louis

13715 Rider Trail North

Earth City, MO 63045

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection, proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2886**

Expiration Date: **6/30/2021**

Effective Date: **7/1/2020**

Sacramento, California
subject to forfeiture or revocation

Christine Sotelo, Chief
Environmental Laboratory Accreditation Program

CALIFORNIA STATE
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

Eurofins TestAmerica St. Louis

13715 Rider Trail North
Earth City, MO 63045
Phone: 3142988566

Certificate No. **2886**
Expiration Date **6/30/2021**

				Primary Accreditation Body
Field of Testing: 106 - Radionuclides in Drinking Water				
106.010	001	Gross Alpha	EPA 900.0	FL
106.010	002	Gross Beta	EPA 900.0	FL
106.030	001	Radioactive Cesium	EPA 901.1	FL
106.050	002	Radium-226	EPA 903.0	FL
106.060	001	Radium-228	EPA 904.0	FL
106.070	003	Strontium-90	EPA 905.0	FL
106.080	001	Tritium	EPA 906.0	FL
106.092	001	Uranium	EPA 200.8	FL
106.220	001	Strontium-89, 90	DOE Sr-01	FL
106.230	001	Uranium	DOE U-02	FL
106.270	001	Gross Alpha	SM 7110 C	FL
106.610	001	Radon-222	SM 7500-Rn	FL
Field of Testing: 108 - Inorganic Constituents in Non-Potable Water				
108.013	001	Calcium	EPA 200.7 (1994 Rev. 4.4)	FL
108.013	002	Magnesium	EPA 200.7 (1994 Rev. 4.4)	FL
108.013	004	Potassium	EPA 200.7 (1994 Rev. 4.4)	FL
108.013	005	Silica, Dissolved	EPA 200.7 (1994 Rev. 4.4)	LA
108.013	006	Sodium	EPA 200.7 (1994 Rev. 4.4)	FL
108.015	001	Calcium	EPA 200.8 (1994 Rev. 5.4)	LA
108.015	002	Magnesium	EPA 200.8 (1994 Rev. 5.4)	FL
108.015	003	Potassium	EPA 200.8 (1994 Rev. 5.4)	LA
108.015	005	Sodium	EPA 200.8 (1994 Rev. 5.4)	FL
108.017	001	Bromide	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	002	Chloride	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	003	Fluoride	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	004	Nitrate (as N)	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	006	Nitrite (as N)	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	007	Phosphate,Ortho (as P)	EPA 300.0 (1993 Rev. 2.1)	FL
108.017	008	Sulfate (as SO4)	EPA 300.0 (1993 Rev. 2.1)	FL
108.137	001	Hydrogen Ion (pH)	SM 4500-H+ B-2011	FL
Field of Testing: 109 - Metals and Trace Elements in Non-Potable Water				
109.623	001	Aluminum	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	002	Antimony	EPA 200.7 (1994 Rev. 4.4)	FL

As of 7/1/2020 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

109.623	003	Arsenic	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	004	Barium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	005	Beryllium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	006	Boron	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	007	Cadmium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	008	Chromium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	009	Cobalt	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	010	Copper	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	011	Iron	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	012	Lead	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	013	Manganese	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	014	Molybdenum	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	015	Nickel	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	016	Selenium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	017	Silver	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	018	Thallium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	019	Tin	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	020	Titanium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	021	Vanadium	EPA 200.7 (1994 Rev. 4.4)	FL
109.623	022	Zinc	EPA 200.7 (1994 Rev. 4.4)	FL
109.625	001	Aluminum	EPA 200.8 (1994 Rev. 5.4)	LA
109.625	002	Antimony	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	003	Arsenic	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	004	Barium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	005	Beryllium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	007	Cadmium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	008	Chromium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	009	Cobalt	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	010	Copper	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	012	Iron	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	013	Lead	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	014	Manganese	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	015	Molybdenum	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	016	Nickel	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	017	Selenium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	018	Silver	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	019	Thallium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	020	Tin	EPA 200.8 (1994 Rev. 5.4)	LA
109.625	021	Titanium	EPA 200.8 (1994 Rev. 5.4)	LA
109.625	022	Vanadium	EPA 200.8 (1994 Rev. 5.4)	FL
109.625	023	Zinc	EPA 200.8 (1994 Rev. 5.4)	FL
109.635	001	Mercury	EPA 245.1 (1994 Rev. 3.0)	FL

Field of Testing: 112 - Radionuclides in Non-Potable Water

112.001	001	Gross Alpha	EPA 900.0		FL
112.001	002	Gross Beta	EPA 900.0		FL
112.003	001	Total Alpha Radium	EPA 903.0		FL

Field of Testing: 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 6010 B		FL
114.010	002	Arsenic	EPA 6010 B		FL
114.010	003	Barium	EPA 6010 B		FL
114.010	004	Beryllium	EPA 6010 B		FL
114.010	005	Cadmium	EPA 6010 B		FL
114.010	006	Chromium	EPA 6010 B		FL
114.010	007	Cobalt	EPA 6010 B		FL
114.010	008	Copper	EPA 6010 B		FL
114.010	009	Lead	EPA 6010 B		FL
114.010	010	Molybdenum	EPA 6010 B		FL
114.010	011	Nickel	EPA 6010 B		FL
114.010	012	Selenium	EPA 6010 B		FL
114.010	013	Silver	EPA 6010 B		FL
114.010	014	Thallium	EPA 6010 B		FL
114.010	015	Vanadium	EPA 6010 B		FL
114.010	016	Zinc	EPA 6010 B		FL
114.020	001	Antimony	EPA 6020		FL
114.020	002	Arsenic	EPA 6020		FL
114.020	003	Barium	EPA 6020		FL
114.020	004	Beryllium	EPA 6020		FL
114.020	005	Cadmium	EPA 6020		FL
114.020	006	Chromium	EPA 6020		FL
114.020	007	Cobalt	EPA 6020		FL
114.020	008	Copper	EPA 6020		FL
114.020	009	Lead	EPA 6020		FL
114.020	010	Molybdenum	EPA 6020		FL
114.020	011	Nickel	EPA 6020		FL
114.020	012	Selenium	EPA 6020		FL
114.020	013	Silver	EPA 6020		FL
114.020	014	Thallium	EPA 6020		FL
114.020	015	Vanadium	EPA 6020		FL
114.020	016	Zinc	EPA 6020		FL
114.103	001	Chromium (VI)	EPA 7196 A	Aqueous Only	FL
114.140	001	Mercury	EPA 7470 A	Aqueous Only	FL
114.141	001	Mercury	EPA 7471 A		FL
114.240	001	Corrosivity - pH Determination	EPA 9040 B	Aqueous Only	FL
114.241	001	Corrosivity - pH Determination	EPA 9045 C		FL

114.250	001	Fluoride	EPA 9056		FL

Field of Testing: 115 - Extraction Test of Hazardous Waste

115.020	001	Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311		FL
115.021	001	TCLP Inorganics	EPA 1311		LA
115.030	001	Waste Extraction Test (WET)	CCR Chapter11, Article 5, Appendix II		LA
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312		FL

Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste

116.030	001	Gasoline-range Organics	EPA 8015 B		FL
116.080	000	Volatile Organic Compounds	EPA 8260 B		FL

Field of Testing: 118 - Radionuclides in Hazardous Waste

118.010	001	Gross Alpha	EPA 9310		FL
118.010	002	Gross Beta	EPA 9310		FL
118.020	001	Radium, Total	EPA 9315		FL
118.030	001	Radium-228	EPA 9320		FL
118.271	001	Strontium	DOE Sr-02		FL
118.290	001	Uranium	DOE U-02		FL

Field of Testing: 120 - Physical Properties of Hazardous Waste

120.070	001	Corrosivity - pH Determination	EPA 9040 B	Aqueous Only	FL
120.080	001	Corrosivity - pH Determination	EPA 9045 C		FL

CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eurofins TestAmerica, St. Louis Facility

13715 Rider Trail North
Earth City, Missouri 63045

Fulfills the requirements of

ISO/IEC 17025:2017

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 06 April 2022
Certificate Number: L2305

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

ANSI National Accreditation Board

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 and U.S. DEPARTMENT OF DEFENSE (DoD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DoD QSM V5.3)

Eurofins TestAmerica, St. Louis Facility
13715 Rider Trail North
Earth City, Missouri 63045
Kristen Ely
314-298-8566

TESTING

Valid to: **April 6, 2022** Certificate Number: **L2305**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Aluminum
ICP-AES	EPA 6010B/6010C/6010D	Antimony
ICP-AES	EPA 6010B/6010C/6010D	Arsenic
ICP-AES	EPA 6010B/6010C/6010D	Barium
ICP-AES	EPA 6010B/6010C/6010D	Beryllium
ICP-AES	EPA 6010B/6010C/6010D	Bismuth
ICP-AES	EPA 6010B/6010C/6010D	Boron
ICP-AES	EPA 6010B/6010C/6010D	Cadmium
ICP-AES	EPA 6010B/6010C/6010D	Calcium
ICP-AES	EPA 6010B/6010C/6010D	Chromium
ICP-AES	EPA 6010B/6010C/6010D	Cobalt
ICP-AES	EPA 6010B/6010C/6010D	Copper
ICP-AES	EPA 6010B/6010C/6010D	Iron
ICP-AES	EPA 6010B/6010C/6010D	Lead
ICP-AES	EPA 6010B/6010C/6010D	Lithium
ICP-AES	EPA 6010B/6010C/6010D	Magnesium
ICP-AES	EPA 6010B/6010C/6010D	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Molybdenum
ICP-AES	EPA 6010B/6010C/6010D	Nickel
ICP-AES	EPA 6010B/6010C/6010D	Phosphorus
ICP-AES	EPA 6010B/6010C/6010D	Potassium
ICP-AES	EPA 6010B/6010C/6010D	Selenium
ICP-AES	EPA 6010B/6010C/6010D	Silicon
ICP-AES	EPA 6010B/6010C/6010D	Silver
ICP-AES	EPA 6010B/6010C/6010D	Sodium
ICP-AES	EPA 6010B/6010C/6010D	Strontium
ICP-AES	EPA 6010B/6010C/6010D	Sulfur
ICP-AES	EPA 6010B/6010C/6010D	Thallium
ICP-AES	EPA 6010B/6010C/6010D	Thorium
ICP-AES	EPA 6010B/6010C/6010D	Tin
ICP-AES	EPA 6010B/6010C/6010D	Titanium
ICP-AES	EPA 6010B/6010C/6010D	Vanadium
ICP-AES	EPA 6010B/6010C/6010D	Zinc
GC/MS	EPA 8260B/8260C/8260D	Acetone
GC/MS	EPA 8260B/8260C/8260D	Acetonitrile
GC/MS	EPA 8260B/8260C/8260D	Acrolein
GC/MS	EPA 8260B/8260C/8260D	Acrylonitrile
GC/MS	EPA 8260B/8260C/8260D	Benzene
GC/MS	EPA 8260B/8260C/8260D	Benzyl chloride
GC/MS	EPA 8260B/8260C/8260D	Bromobenzene
GC/MS	EPA 8260B/8260C/8260D	Bromochloromethane
GC/MS	EPA 8260B/8260C/8260D	Bromodichloromethane
GC/MS	EPA 8260B/8260C/8260D	Bromoform
GC/MS	EPA 8260B/8260C/8260D	Bromomethane
GC/MS	EPA 8260B/8260C/8260D	n-Butanol
GC/MS	EPA 8260B/8260C/8260D	2-Butanone
GC/MS	EPA 8260B/8260C/8260D	n-Butylbenzene
GC/MS	EPA 8260B/8260C/8260D	sec-Butylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	tert-Butylbenzene
GC/MS	EPA 8260B/8260C/8260D	Carbon disulfide
GC/MS	EPA 8260B/8260C/8260D	Carbon tetrachloride
GC/MS	EPA 8260B/8260C/8260D	Chlorobenzene
GC/MS	EPA 8260B/8260C/8260D	2-Chloro-1,3-butadiene
GC/MS	EPA 8260B/8260C/8260D	Chlorodibromomethane
GC/MS	EPA 8260B/8260C/8260D	Chloroethane
GC/MS	EPA 8260B/8260C/8260D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/8260C/8260D	Chloroform
GC/MS	EPA 8260B/8260C/8260D	Chloromethane
GC/MS	EPA 8260B/8260C/8260D	Allyl chloride
GC/MS	EPA 8260B/8260C/8260D	2-Chlorotoluene
GC/MS	EPA 8260B/8260C/8260D	4-Chlorotoluene
GC/MS	EPA 8260B/8260C/8260D	Cyclohexane
GC/MS	EPA 8260B/8260C/8260D	Cyclohexanone
GC/MS	EPA 8260B/8260C/8260D	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C/8260D	Dibromomethane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/8260C/8260D	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C/8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloroethene (total)
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C/8260D	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C/8260D	2,2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 8260B/8260C/8260D	Dimethyl disulfide
GC/MS	EPA 8260B/8260C/8260D	1,4-Dioxane
GC/MS	EPA 8260B/8260C/8260D	Ethyl acetate
GC/MS	EPA 8260B/8260C/8260D	Ethylbenzene
GC/MS	EPA 8260B/8260C/8260D	Ethyl ether
GC/MS	EPA 8260B/8260C/8260D	Ethyl methacrylate
GC/MS	EPA 8260B/8260C/8260D	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C/8260D	n-Hexane
GC/MS	EPA 8260B/8260C/8260D	2-Hexanone
GC/MS	EPA 8260B/8260C/8260D	Iodomethane
GC/MS	EPA 8260B/8260C/8260D	Isobutanol
GC/MS	EPA 8260B/8260C/8260D	Isopropylbenzene
GC/MS	EPA 8260B/8260C/8260D	p-Isopropyltoluene
GC/MS	EPA 8260B/8260C/8260D	Methacrylonitrile
GC/MS	EPA 8260B/8260C/8260D	Methyl acetate
GC/MS	EPA 8260B/8260C/8260D	Methylcyclohexane
GC/MS	EPA 8260B/8260C/8260D	Methylene chloride
GC/MS	EPA 8260B/8260C/8260D	Methyl methacrylate
GC/MS	EPA 8260B/8260C/8260D	4-Methyl-2-pentanone
GC/MS	EPA 8260B/8260C/8260D	MTBE
GC/MS	EPA 8260B/8260C/8260D	Naphthalene
GC/MS	EPA 8260B/8260C/8260D	2-Nitropropane
GC/MS	EPA 8260B/8260C/8260D	Nonanal
GC/MS	EPA 8260B/8260C/8260D	Pentachloroethane
GC/MS	EPA 8260B/8260C/8260D	Propionitrile
GC/MS	EPA 8260B/8260C/8260D	n-Propylbenzene
GC/MS	EPA 8260B/8260C/8260D	Styrene
GC/MS	EPA 8260B/8260C/8260D	1,1,1,2-Tetrachloroethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C/8260D	Tetrachloroethene
GC/MS	EPA 8260B/8260C/8260D	Tetrahydrofuran
GC/MS	EPA 8260B/8260C/8260D	Toluene
GC/MS	EPA 8260B/8260C/8260D	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C/8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C/8260D	Trichloroethene
GC/MS	EPA 8260B/8260C/8260D	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C/8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C/8260D	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C/8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C/8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C/8260D	Vinyl acetate
GC/MS	EPA 8260B/8260C/8260D	Vinyl chloride
GC/MS	EPA 8260B/8260C/8260D	m-Xylene & p-Xylene
GC/MS	EPA 8260B/8260C/8260D	o-Xylene
GC/MS	EPA 8260B/8260C/8260D	Xylenes (total)
GC/MS	EPA 8260B/8260C/8260D SIM	1,4-Dioxane
ICP-MS	EPA 6020/6020A/6020B	Aluminum
ICP-MS	EPA 6020/6020A/6020B	Antimony
ICP-MS	EPA 6020/6020A/6020B	Arsenic
ICP-MS	EPA 6020/6020A/6020B	Barium
ICP-MS	EPA 6020/6020A/6020B	Beryllium
ICP-MS	EPA 6020/6020A/6020B	Bismuth
ICP-MS	EPA 6020/6020A/6020B	Boron
ICP-MS	EPA 6020/6020A/6020B	Cadmium
ICP-MS	EPA 6020/6020A/6020B	Calcium
ICP-MS	EPA 6020/6020A/6020B	Cerium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Cesium
ICP-MS	EPA 6020/6020A/6020B	Chromium
ICP-MS	EPA 6020/6020A/6020B	Cobalt
ICP-MS	EPA 6020/6020A/6020B	Copper
ICP-MS	EPA 6020/6020A/6020B	Gold
ICP-MS	EPA 6020/6020A/6020B	Hafnium
ICP-MS	EPA 6020/6020A/6020B	Iron
ICP-MS	EPA 6020/6020A/6020B	Lanthanum
ICP-MS	EPA 6020/6020A/6020B	Lead
ICP-MS	EPA 6020/6020A/6020B	Lithium
ICP-MS	EPA 6020/6020A/6020B	Magnesium
ICP-MS	EPA 6020/6020A/6020B	Manganese
ICP-MS	EPA 6020/6020A/6020B	Molybdenum
ICP-MS	EPA 6020/6020A/6020B	Neodymium
ICP-MS	EPA 6020/6020A/6020B	Nickel
ICP-MS	EPA 6020/6020A/6020B	Niobium
ICP-MS	EPA 6020/6020A/6020B	Palladium
ICP-MS	EPA 6020/6020A/6020B	Phosphorus
ICP-MS	EPA 6020/6020A/6020B	Platinum
ICP-MS	EPA 6020/6020A/6020B	Potassium
ICP-MS	EPA 6020/6020A/6020B	Praseodymium
ICP-MS	EPA 6020/6020A/6020B	Rhenium
ICP-MS	EPA 6020/6020A/6020B	Rhodium
ICP-MS	EPA 6020/6020A/6020B	Ruthenium
ICP-MS	EPA 6020/6020A/6020B	Samarium
ICP-MS	EPA 6020/6020A/6020B	Selenium
ICP-MS	EPA 6020/6020A/6020B	Silver
ICP-MS	EPA 6020/6020A/6020B	Sodium
ICP-MS	EPA 6020/6020A/6020B	Strontium
ICP-MS	EPA 6020/6020A/6020B	Tantalum
ICP-MS	EPA 6020/6020A/6020B	Tellurium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Thallium
ICP-MS	EPA 6020/6020A/6020B	Thorium
ICP-MS	EPA 6020/6020A/6020B	Tin
ICP-MS	EPA 6020/6020A/6020B	Titanium
ICP-MS	EPA 6020/6020A/6020B	Tungsten
ICP-MS	EPA 6020/6020A/6020B	Uranium
ICP-MS	EPA 6020/6020A/6020B	Uranium 233
ICP-MS	EPA 6020/6020A/6020B	Uranium 234
ICP-MS	EPA 6020/6020A/6020B	Uranium 235
ICP-MS	EPA 6020/6020A/6020B	Uranium 236
ICP-MS	EPA 6020/6020A/6020B	Uranium 238
ICP-MS	EPA 6020/6020A/6020B	Vanadium
ICP-MS	EPA 6020/6020A/6020B	Yttrium
ICP-MS	EPA 6020/6020A/6020B	Zinc
ICP-MS	EPA 6020/6020A/6020B	Zirconium
ICP-MS	EPA 6020/6020A/6020B	Total Hardness
ICP-MS	EPA 6020/6020A/6020B	Dysprosium
ICP-MS	EPA 6020/6020A/6020B	Erbium
ICP-MS	EPA 6020/6020A/6020B	Europium
ICP-MS	EPA 6020/6020A/6020B	Gadolinium
ICP-MS	EPA 6020/6020A/6020B	Gallium
ICP-MS	EPA 6020/6020A/6020B	Holmium
ICP-MS	EPA 6020/6020A/6020B	Lutetium
ICP-MS	EPA 6020/6020A/6020B	Rubidium
ICP-MS	EPA 6020/6020A/6020B	Terbium
ICP-MS	EPA 6020/6020A/6020B	Thulium
ICP-MS	EPA 6020/6020A/6020B	Ytterbium
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Cerium
ICP-MS	EPA 200.8	Cesium
ICP-MS	EPA 200.8	Chromium
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Gold
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Phosphorus
ICP-MS	EPA 200.8	Platinum
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Rhodium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium
ICP-MS	EPA 200.8	Tantalum
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Thorium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Tungsten

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium
ICP-MS	EPA 200.8	Zinc
ICP-MS	EPA 200.8	Zirconium
ICP-AES	EPA 200.7	Aluminum
ICP-AES	EPA 200.7	Antimony
ICP-AES	EPA 200.7	Arsenic
ICP-AES	EPA 200.7	Barium
ICP-AES	EPA 200.7	Beryllium
ICP-AES	EPA 200.7	Bismuth
ICP-AES	EPA 200.7	Boron
ICP-AES	EPA 200.7	Cadmium
ICP-AES	EPA 200.7	Calcium
ICP-AES	EPA 200.7	Chromium
ICP-AES	EPA 200.7	Cobalt
ICP-AES	EPA 200.7	Copper
ICP-AES	EPA 200.7	Iron
ICP-AES	EPA 200.7	Lead
ICP-AES	EPA 200.7	Lithium
ICP-AES	EPA 200.7	Magnesium
ICP-AES	EPA 200.7	Manganese
ICP-AES	EPA 200.7	Molybdenum
ICP-AES	EPA 200.7	Nickel
ICP-AES	EPA 200.7	Phosphorus
ICP-AES	EPA 200.7	Potassium
ICP-AES	EPA 200.7	Selenium
ICP-AES	EPA 200.7	Silicon
ICP-AES	EPA 200.7	Silver
ICP-AES	EPA 200.7	Sodium
ICP-AES	EPA 200.7	Strontium
ICP-AES	EPA 200.7	Sulfur

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 200.7	Thallium
ICP-AES	EPA 200.7	Thorium
ICP-AES	EPA 200.7	Tin
ICP-AES	EPA 200.7	Titanium
ICP-AES	EPA 200.7	Vanadium
ICP-AES	EPA 200.7	Zinc
CVAA	EPA 245.1/7470A	Mercury
Ion Chromatrography	EPA 300.0/9056/9056A	Bromide
Ion Chromatrography	EPA 300.0/9056/9056A	Chloride
Ion Chromatrography	EPA 300.0/9056/9056A	Fluoride
Ion Chromatrography	EPA 300.0/9056/9056A	Nitrate
Ion Chromatrography	EPA 300.0/9056/9056A	Nitrite
Ion Chromatrography	EPA 300.0/9056/9056A	Sulfate
Ion Chromatrography	EPA 300.0/9056/9056A	Ortho-phosphate
Ion Chromatrography	EPA 300.0/9056/9056A	Iodide
Probe	EPA 9040C EPA 150.1 SM 4500-H+ B -2011	pH
Colormetric	EPA 7196A	Hex Chromium
Gas Flow Proportional Counter	EPA 900.0 EPA 9310 SM 7110C	gross alpha/beta
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02 DOE HASL 300 Sr-03	Strontium-90
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222

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Non-Potable Water		
Technology	**Method**	**Analyte**
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01 HASL 300 Tc-02-RC	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212

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Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46

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Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R DOE HASL 300 U-02-RC	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium

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Non-Potable Water		
Technology	**Method**	**Analyte**
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Alpha Spectroscopy	ST-RC-0301	Radium-226
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Extraction Chromatography	ST-RC-0058	Strontium-90
Preparation	**Method**	**Type**
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Acid Digestion (Aqueous samples)	EPA 3010A EPA 3005A	Acid Digestion for Metals (Aqueous samples)
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction

Drinking Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Uranium
Alpha Spectroscopy	DOE HASL 300 U-02-RC	Isotopic Uranium
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	Gross alpha/beta
Gas Flow Proportional Counter	SM 7110C	Gross alpha

Drinking Water		
Technology	**Method**	**Analyte**
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Aluminum
ICP-AES	EPA 6010B/6010C/6010D	Antimony
ICP-AES	EPA 6010B/6010C/6010D	Arsenic
ICP-AES	EPA 6010B/6010C/6010D	Barium
ICP-AES	EPA 6010B/6010C/6010D	Beryllium
ICP-AES	EPA 6010B/6010C/6010D	Bismuth
ICP-AES	EPA 6010B/6010C/6010D	Boron
ICP-AES	EPA 6010B/6010C/6010D	Cadmium
ICP-AES	EPA 6010B/6010C/6010D	Calcium
ICP-AES	EPA 6010B/6010C/6010D	Chromium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Cobalt
ICP-AES	EPA 6010B/6010C/6010D	Copper
ICP-AES	EPA 6010B/6010C/6010D	Iron
ICP-AES	EPA 6010B/6010C/6010D	Lead
ICP-AES	EPA 6010B/6010C/6010D	Lithium
ICP-AES	EPA 6010B/6010C/6010D	Magnesium
ICP-AES	EPA 6010B/6010C/6010D	Manganese
ICP-AES	EPA 6010B/6010C/6010D	Molybdenum
ICP-AES	EPA 6010B/6010C/6010D	Nickel
ICP-AES	EPA 6010B/6010C/6010D	Phosphorus
ICP-AES	EPA 6010B/6010C/6010D	Potassium
ICP-AES	EPA 6010B/6010C/6010D	Selenium
ICP-AES	EPA 6010B/6010C/6010D	Silicon
ICP-AES	EPA 6010B/6010C/6010D	Silver
ICP-AES	EPA 6010B/6010C/6010D	Sodium
ICP-AES	EPA 6010B/6010C/6010D	Strontium
ICP-AES	EPA 6010B/6010C/6010D	Sulfur
ICP-AES	EPA 6010B/6010C/6010D	Thallium
ICP-AES	EPA 6010B/6010C/6010D	Thorium
ICP-AES	EPA 6010B/6010C/6010D	Tin
ICP-AES	EPA 6010B/6010C/6010D	Titanium
ICP-AES	EPA 6010B/6010C/6010D	Vanadium
ICP-AES	EPA 6010B/6010C/6010D	Zinc
ICP-MS	EPA 6020/6020A/6020B	Aluminum
ICP-MS	EPA 6020/6020A/6020B	Antimony
ICP-MS	EPA 6020/6020A/6020B	Arsenic
ICP-MS	EPA 6020/6020A/6020B	Barium
ICP-MS	EPA 6020/6020A/6020B	Beryllium
ICP-MS	EPA 6020/6020A/6020B	Bismuth
ICP-MS	EPA 6020/6020A/6020B	Boron
ICP-MS	EPA 6020/6020A/6020B	Cadmium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Calcium
ICP-MS	EPA 6020/6020A/6020B	Cerium
ICP-MS	EPA 6020/6020A/6020B	Cesium
ICP-MS	EPA 6020/6020A/6020B	Chromium
ICP-MS	EPA 6020/6020A/6020B	Cobalt
ICP-MS	EPA 6020/6020A/6020B	Copper
ICP-MS	EPA 6020/6020A/6020B	Gold
ICP-MS	EPA 6020/6020A/6020B	Hafnium
ICP-MS	EPA 6020/6020A/6020B	Iron
ICP-MS	EPA 6020/6020A/6020B	Lanthanum
ICP-MS	EPA 6020/6020A/6020B	Lead
ICP-MS	EPA 6020/6020A/6020B	Lithium
ICP-MS	EPA 6020/6020A/6020B	Magnesium
ICP-MS	EPA 6020/6020A/6020B	Manganese
ICP-MS	EPA 6020/6020A/6020B	Molybdenum
ICP-MS	EPA 6020/6020A/6020B	Neodymium
ICP-MS	EPA 6020/6020A/6020B	Nickel
ICP-MS	EPA 6020/6020A/6020B	Niobium
ICP-MS	EPA 6020/6020A/6020B	Palladium
ICP-MS	EPA 6020/6020A/6020B	Phosphorus
ICP-MS	EPA 6020/6020A/6020B	Platinum
ICP-MS	EPA 6020/6020A/6020B	Potassium
ICP-MS	EPA 6020/6020A/6020B	Praseodymium
ICP-MS	EPA 6020/6020A/6020B	Rhenium
ICP-MS	EPA 6020/6020A/6020B	Rhodium
ICP-MS	EPA 6020/6020A/6020B	Ruthenium
ICP-MS	EPA 6020/6020A/6020B	Samarium
ICP-MS	EPA 6020/6020A/6020B	Selenium
ICP-MS	EPA 6020/6020A/6020B	Silver
ICP-MS	EPA 6020/6020A/6020B	Sodium
ICP-MS	EPA 6020/6020A/6020B	Strontium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Tantalum
ICP-MS	EPA 6020/6020A/6020B	Tellurium
ICP-MS	EPA 6020/6020A/6020B	Thallium
ICP-MS	EPA 6020/6020A/6020B	Thorium
ICP-MS	EPA 6020/6020A/6020B	Tin
ICP-MS	EPA 6020/6020A/6020B	Titanium
ICP-MS	EPA 6020/6020A/6020B	Tungsten
ICP-MS	EPA 6020/6020A/6020B	Uranium
ICP-MS	EPA 6020/6020A/6020B	Uranium 233
ICP-MS	EPA 6020/6020A/6020B	Uranium 234
ICP-MS	EPA 6020/6020A/6020B	Uranium 235
ICP-MS	EPA 6020/6020A/6020B	Uranium 236
ICP-MS	EPA 6020/6020A/6020B	Uranium 238
ICP-MS	EPA 6020/6020A/6020B	Vanadium
ICP-MS	EPA 6020/6020A/6020B	Yttrium
ICP-MS	EPA 6020/6020A/6020B	Zinc
ICP-MS	EPA 6020/6020A/6020B	Zirconium
CVAA	EPA 7471A/7471B	Mercury
Ion Chromatrography	EPA 300.0/9056A	Bromide
Ion Chromatrography	EPA 300.0/9056A	Chloride
Ion Chromatrography	EPA 300.0/9056A	Fluoride
Ion Chromatrography	EPA 300.0/9056A	Nitrate
Ion Chromatrography	EPA 300.0/9056A	Nitrite
Ion Chromatrography	EPA 300.0/9056A	Sulfate
Ion Chromatrography	EPA 300.0/9056A	Ortho-phosph
Ion Chromatrography	EPA 300.0/9056A	Iodide
Probe	EPA 9045D	pH
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02 DOE HASL 300 Sr-03	Strontium-90
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01 HASL 300 Tc-02-RC	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R DOE HASL 300 U-02-RC	Isotopic Uranium

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Alpha Spectroscopy	ST-RC-0301	Radium-226
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Preparation	**Method**	**Type**
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Acid Digestion (solids)	EPA 3050B	Acid Digestion for Metals of Sedimtent/Soils
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile Samples
Closed System Purge & Trap and Extraction for Volatiles	EPA 5035A	Closed System Purge & Trap and Extraction for Volatiles
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Extraction Chromatography	Eichrom Technologies FEW01	Iron-55
Extraction Chromatography	ST-RC-0058	Strontium-90

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2305.

R. Douglas Leonard Jr., VP, PILR SBU

NRC FORM 374

U.S. NUCLEAR REGULATORY COMMISSION

Amendment No. 25

MATERIALS LICENSE

Pursuant to the Atomic Energy Act of 1954, as amended, the Energy Reorganization Act of 1974 (Public Law 93-438), and Title 10, Code of Federal Regulations, Chapter I, Parts 30, 31, 32, 33, 34, 35, 36, 37, 39, 40, 70 and 71, and in reliance on statements and representations heretofore made by the licensee, a license is hereby issued authorizing the licensee to receive, acquire, possess, and transfer byproduct, source, and special nuclear material designated below; to use such material for the purpose(s) and at the place(s) designated below; to deliver or transfer such material to persons authorized to receive it in accordance with the regulations of the applicable Part(s). This license shall be deemed to contain the conditions specified in Section 183 of the Atomic Energy Act of 1954, as amended, and is subject to all applicable rules, regulations, and orders of the Nuclear Regulatory Commission now or hereafter in effect and to any conditions specified below.

Licensee	In accordance with letter dated November 09, 2018.	4. Expiration Date: December 31, 2022
1. TestAmerica Laboratories, Inc.		5. Docket No.: 030-29601 Reference No.:
2. 13715 Rider Trail North Earth City, MO 63045	3. License number: 24-24817-01 is amended in its entirety to read as follows:	

6. Byproduct, source, and/or special nuclear material	7. Chemical and/or physical form	8. Maximum amount that licensee may possess at any one time under this license	9. Authorized use
A. Any byproduct material specified in section 33.100, Schedule A, with exceptions	A. Any	A. As specified in 10 CFR Part 33, Schedule A, Column II	A. For use and/or possession incident to the performance of chemical and radiochemical analysis of samples and as calibration and/or reference standards in performing instrument calibration.
B. Strontium-90	B. Any	B. 100 millicuries total	B. Same as Subitem No. 9.A.
C. Cesium-137	C. Any	C. 100 millicuries total	C. Same as Subitem No. 9.A.
D. Cobalt-60	D. Any	D. 100 millicuries total	D. Same as Subitem No. 9.A.
E. Hydrogen-3	E. Any	E. 3 curies total	E. Same as Subitem No. 9.A.
F. Thorium (Natural)	F. Any	F. 200 millicuries total	F. Same as Subitem No. 9.A.
G. Uranium-233	G. Any	G. 19 millicuries (2 grams) total	G. Same as Subitem No. 9.A.

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MATERIALS LICENSE SUPPLEMENTARY SHEET

License Number: 24-24817-01
Amendment No. 25

Docket or Reference Number: 030-29601

6. Byproduct, source, and/or special nuclear material	7. Chemical and/or physical form	8. Maximum amount that licensee may possess at any one time under this license	9. Authorized use
H. Uranium-235-irradiated and unirradiated	H. Any	H. 714 microcuries (340 grams) total	H. Same as Subitem No. 9.A.
I. Any byproduct material with Atomic Numbers 84 through 103	I. Any	I. 5 millicuries per radionuclide and 100 millicuries total	I. Same as Subitem No. 9.A.
J. Uranium (Natural)	J. Any	J. 136 kilograms total	J. Same as Subitem No. 9.A.
K. Plutonium-236	K. Any	K. 100 microcuries total	K. Same as Subitem No. 9.A.
L. Plutonium-238	L. Any	L. 100 microcuries total	L. Same as Subitem No. 9.A.
M. Plutonium-239	M. Any	M. 100 microcuries total	M. Same as Subitem No. 9.A.
N. Plutonium-240	N. Any	N. 100 microcuries total	N. Same as Subitem No. 9.A.
O. Plutonium-241	O. Any	O. 5 millicuries total	O. Same as Subitem No. 9.A.
P. Plutonium-242	P. Any	P. 100 microcuries total	P. Same as Subitem No. 9.A.
Q. Thorium-227	Q. Any	Q. 2.1 millicuries total	Q. Same as Subitem No. 9.A.
R. Thorium-228	R. Any	R. 200 millicuries total	R. Same as Subitem No. 9.A.
S. Thorium-229	S. Any	S. 100 microcuries total	S. Same as Subitem No. 9.A.
T. Thorium-230	T. Any	T. 45.4 millicuries total	T. Same as Subitem No. 9.A.
U. Thorium-231	U. Any	U. 2.1 millicuries total	U. Same as Subitem No. 9.A.
V. Thorium-232	V. Any	V. 200 millicuries total	V. Same as Subitem No. 9.A.
W. Thorium-234	W. Any	W. 45.4 millicuries total	W. Same as Subitem No. 9.A.

UNITED STATES NUCLEAR REGULATORY COMMISSION

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MATERIALS LICENSE SUPPLEMENTARY SHEET

License Number: 24-24817-01
Amendment No. 25

Docket or Reference Number: 030-29601

6. Byproduct, source, and/or special nuclear material	7. Chemical and/or physical form	8. Maximum amount that licensee may possess at any one time under this license	9. Authorized use
X. Barium-133	X. Sealed Sources	X. 8 sources; not to exceed 20 microcuries per source; 160 microcuries total	X. For use as internal calibration sources for liquid scintillation counters.
Y. Nickel-63	Y. Foils or plated sources	Y. 21 sources; not to exceed 15 millicuries per source; 315 millicuries total	Y. For use of foils or plated sources, registered either with NRC under 10 CFR 32.210 or with an Agreement State, and incorporated in compatible Agilent (formerly Hewlett-Packard) Model 6890 gas chromatography devices for sample analysis.

CONDITIONS

10. Licensed material may be used or stored at the licensee's facilities located at: 13715 Rider Trail North, Earth City, Missouri, 63045

11. The Radiation Safety Officer (RSO) for this license is Michael Ridenhower.

12. Licensed material shall only be used by, or under the supervision of, individuals listed below, for materials and uses as indicated:

Authorized Individuals	Material and Use
Michael Ridenhower	All
Terrance Romanko	All

13. In addition to the possession limits in Item 8, the licensee shall further restrict the possession of licensed material to quantities below the limits specified in 10 CFR 30.72 which require consideration of the need for an emergency plan for responding to a release of licensed material.

ED_006787_00009423-00215

MATERIALS LICENSE SUPPLEMENTARY SHEET	License Number 24-24817-01	Docket or Reference Number 030-29601
	Amendment No. 25	

14. A. Sealed sources and detector cells shall be tested for leakage and/or contamination at intervals not to exceed the intervals specified in the certificate of registration issued by the U.S. Nuclear Regulatory Commission under 10 CFR 32.210 or by an Agreement State. In the absence of a registration certificate, sealed sources shall be tested for leakage and/or contamination at intervals not to exceed 6 months, or at such other intervals as specified.

B. In the absence of a certificate from a transferor indicating that a leak test has been made within the intervals specified in the certificate of registration issued by the U.S. Nuclear Regulatory Commission under 10 CFR 32.210 or by an Agreement State, prior to the transfer, a sealed source received from another person shall not be put into use until tested and the test results received.

C. Sealed sources need not be tested if they contain only hydrogen-3; or they contain only a radioactive gas; or the half-life of the isotope is 30 days or less; or they contain not more than 100 microcuries of beta- and/or gamma-emitting material or not more than 10 microcuries of alpha-emitting material.

D. Sealed sources need not be tested if they are in storage and are not being used. However, when they are removed from storage for use or transferred to another person, and have not been tested within the required leak test interval, they shall be tested before use or transfer. No sealed source shall be stored for a period of more than 10 years without being tested for leakage and/or contamination.

E. The leak test shall be capable of detecting the presence of 185 becquerels (0.005 microcuries) of radioactive material on the test sample. If the test reveals the presence of 185 becquerels (0.005 microcuries) or more of removable contamination, a report shall be filed with the U.S. Nuclear Regulatory Commission in accordance with 10 CFR 30.50(c)(2), and the source shall be removed immediately from service and decontaminated, repaired, or disposed of in accordance with Commission regulations.

F. Tests for leakage and/or contamination, including leak test sample collection and analysis, shall be performed by the licensee or other persons specifically licensed by the U.S. Nuclear Regulatory Commission or an Agreement State to perform such services.

G. Records of leak test results shall be kept in units of becquerels (microcuries) and shall be maintained for 3 years.

ED_006787_00009423-00216

MATERIALS LICENSE SUPPLEMENTARY SHEET	License Number 24-24817-01	Docket or Reference Number 030-29601
	Amendment No. 25	

15. The licensee shall conduct a physical inventory every 6 months, or at other intervals approved by the U.S. Nuclear Regulatory Commission, to account for all sealed sources and/or devices received and possessed under the license. Records of inventories shall be maintained for 3 years from the date of each inventory, and shall include the radionuclides, quantities, manufacturer's name and model numbers, and the date of the inventory.

16. Sealed sources or detector cells containing licensed material shall not be opened or sources removed from source holders or foil sources removed from detector cells by the licensee, except as specifically authorized.

17. The licensee shall not use the licensed material in or on humans.

18. The licensee shall not acquire licensed material in a sealed source or device unless the source or device has been registered with the U.S. Nuclear Regulatory Commission pursuant to 10 CFR 32.210 or equivalent regulations of an Agreement State.

19. Maintenance, repair, cleaning, replacement, and disposal of foils contained in detector cells shall be performed only by the device manufacturer or other persons specifically authorized by the U.S Nuclear Regulatory Commission or an Agreement State to perform such services.

20. Except as specifically provided otherwise in this license, the licensee shall conduct its program in accordance with the statements, representations, and procedures contained in the documents, including any enclosures, listed below. This license condition applies only to those procedures that are required to be submitted in accordance with the regulations. The U.S. Nuclear Regulatory Commission's regulations shall govern unless the statements, representations, and procedures in the licensee's application and correspondence are more restrictive than the regulations.

    A. Application dated June 29, 2012 (ML12187A196)
    B. Letter dated November 28, 2012 (ML12333A358)
    C. Letter dated July 26, 2013 (ML13211A488)
    D. Letter dated September 28, 2016 (ML16279A470)

ED_006787_00009423-00217

MATERIALS LICENSE SUPPLEMENTARY SHEET	License Number 24-24817-01	Docket or Reference Number 030-29601
	Amendment No. 25	

E. Letter dated April 12, 2017 (ML17018A411)
F. Letter dated April 1, 2019 (ML19092A369)

Date: May 13, 2019

FOR THE U.S. NUCLEAR REGULATORY COMMISSION

By: Sara A. Forster
Sara A. Forster
Region 3

ED_006787_00009423-00218

Title: DRYING AND GRINDING OF SOIL AND SOLID SAMPLES for RADIOCHEMISTRY ANALYSIS

Approvals (Signature/Date):

Mike Aldridge — Date: 3-27-19 Pre-Prep Supervisor	Michael Ridenhower — Date: 3/20/19 Health & Safety Manager / Coordinator
Kristen Ely — Date: 3/27/19 Quality Assurance Manager	Andrew Buettner — Date: 3-20-19 Operations Manager

This SOP was previously identified as SOP No. ST-RC-0003 Rev. 16

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1.0 SCOPE AND APPLICATION

1.1 This procedure describes the method for drying, reducing particle size and homogenizing samples for radiochemical analysis.

1.2 This method is applicable to solid samples including sediment and soil.

1.3 This method is not applicable to solid samples such as oils, filters, fauna, flora and certain types of "destruction and debris" materials. Consult the radiochemistry manager or supervisor to determine if the "destruction and debris" material is applicable to this procedure.

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for drying and grinding samples to a uniform particle size so that radiochemical analyses can be performed.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 Rolling Rocks –inert grinding cylinders used to homogenize samples on the ball mill.

4.0 INTERFERENCES

4.1 This method may cause evaporation of volatile radionuclides such as iodine, tritium and carbon-14. This method is not to be used for samples that require volatile chemical analysis.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
Hearing protection is required when using the puck-mill or ball mill in the event that noise levels exceed 90 dB(A) within 3 feet of the grinder. Contact the EH&S Coordinator to determine the results of the noise surveys.

5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Compound	Hazards	Exposure Limits (1)	Signs of Exposure
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Methanol (Methyl Alcohol)	Poison Flammable	200 ppm (TWA)	Inhalation symptoms may include headache, drowsiness, nausea, vomiting, blurred vision and blindness. Symptoms of skin contact may parallel inhalation exposure and can cause skin to become dry and cracked. Eye contact can cause irritation. Vapor harmful. Harmful if inhaled or absorbed through skin. Flammable liquid and vapor. Causes irritation to skin, eyes and respiratory tract.
1- Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short term exposure limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Ball mill

6.2 Puck-mill (pulverizer), including dish and puck

6.3 Analytical Balance

6.4 Aluminum pans (small and large)

6.5 Drying Oven

6.6 GM 44-9 beta/gamma probe (frisker)

6.7 Cooling rack

6.8 Paint cans with lids, ½ gallon and 1 gallon

6.9 Duct tape (or equivalent)

6.10 Rolling Rocks

6.11 Large paint brush (for cleaning balance)

6.12 Bucket

6.13 5 gallon cooler with spout

6.14 4 – mesh sieve (Various mesh sieves as methods require)

6.15 Mortar and Pestle (for air filters)

6.16 Various size/types of containers, including but not limited to:

6.16.1 0.5 L and 1 L Marinelli beaker

6.16.2 500 mL and 1 L wide-mouth jar

6.16.3 25 mL and 100 mL containers

6.17 Additional items needed for Coning & Quartering

6.17.1 Wax paper
6.17.2 Flat press
6.17.3 Splitting tool

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Quartz sand

7.3 Deionized water

7.4 Methanol

7.5 10% radiac solution – for every 1L of water, 100mL of Radiac is required.

7.6 10% bleach solution – for every 1L of water 100mL of bleach is required.

7.7 Nitric Acid (concentrated [16N] and 1:1 [8N])

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods cited and other applicable sample collection documents for detailed collection procedures. Sample volume and preservative information is given in ST-PM-0002.

8.2 Soil samples may be collected in either plastic or glass containers.

9.0 QUALITY CONTROL

9.1 **Decontamination Blank (Pulverizer)**

9.1.1 A pulverizer decontamination rinse blank is analyzed at the end of every 20 samples or completion of the batch.
9.1.2 The pulverizer decontamination blank consists of acidified DI water.
9.1.3 See section 11.6 of this SOP for cleaning and collecting of pulverizer decontamination blanks.
9.1.4 The pulverizer decontamination blank is processed for gamma and gross alpha beta.
9.1.5 The pass/fail criteria for rolling rocks are < MDC

9.2 **Decontamination Blank (Ball Mill)**

9.2.1 A Ball Mill (rolling rocks) decontamination rinsate blank is analyzed as needed after cleaning the rolling rocks.
9.2.2 The rolling rock decontamination blank consists of acidified DI water.
9.2.3 See section 11.4 of this SOP for cleaning and collecting of rolling rocks decontamination rinsate blank.
9.2.4 The rolling rock decontamination blank is processed for gamma and gross alpha beta.
9.2.5 The pass/fail criteria for rolling rocks are < MDC

9.3 **Procedural Variations/ Nonconformance and Corrective Action**

9.3.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.3.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 There are no calibration requirements for this preparation procedure.

11.0 PROCEDURE

11.1 **Sizing of Sample**

11.1.1 For solid samples with various particle sizes, including pebbles or small rocks, processing by ball milling may be adequate to achieve a uniform particle size.
11.1.2 For solid samples that must be reduced to a fine mesh size (i.e. 200 mesh), a pulverizer will be necessary.
11.1.3 For solid samples that are composed of large particles such as rock, cement, stone, etc., it may be necessary to fracture or reduce the size of the sample before pulverizing.
11.1.4 Refer to method comments to determine if there are client specific sizing requirements.
11.1.5 In an effort to avoid cross contamination, the order of the samples processed should be from low activity to high radiological activity, if the sample activty is known. Screening results or surveying with a GM 44-9 beta/gamma probe may be used for this purpose.

11.2 **Drying of Samples - Non-volatile Isotopes**

NOTE: If entire sample is to be dried: Prior to drying the sample, the requested analyte list must be evaluated for volatile components. Not all analytes are amenable to the drying process and severe losses may result from the drying. If there is uncertainty on this point, seek technical assistance prior to drying.

11.2.1 Place sample in labeled, aluminum pan. Frisk sample with GM 44-9 beta/gamma probe.
11.2.1.1 The amount of sample dried must be sufficient to cover the minimum sample amounts needed for the analyses requested. See ST-PM-0002 for minimum sample amounts.
11.2.1.2 If drying of the entire sample is required by the client, multiple pans may be necessary.
11.2.2 Frisk all samples with a GM 44-9 beta/gamma probe.
11.2.2.1 Any sample reading 100 cpm over background will need a red dot sticker (indicating radioactivity) on the side of the bread pan. .
11.2.3 Place sample in a drying oven for at least 8 hours at approximately 105°C +/- 5°C. Record time, date, lot and initials in the Dry/Grind Logbook (Attachment 1)
11.2.3.1 Visually inspect the sample at the end of the drying period. If the sample does not appear to be dry, return sample to oven to continue drying.
11.2.3.2 When the sample is dry, remove the sample from the oven and place on cooling rack (or other designated spot). Record the oven temperature, date, time and analyst initials in the Dry/Grind Logbook.
11.2.3.3 Allow the sample to cool to room temperature.

11.3 **Ball Mill Procedure**

11.3.1 Start with dried sample as specified in the Drying section of this procedure.
11.3.2 Print a label for the paint can and place the label on the side of the can. The sample ID may also be written in permanent black marker on the lid of the paint can if deemed beneficial. Load dried sample into can.

11.3.2.1 If the drying pan was labeled with a red dot sticker, place one on the paint can next to the ID label. Place another red dot sticker on the lid (in case the sticker on the side comes off)

11.3.2.2 Do not fill the paint can over halfway full. If the can is filled too much, inefficient grinding will occur.

11.3.2.3 If grinding of entire sample is required, multiple paint cans may be necessary. Label each can appropriately.

11.3.3 Place 3-5 rolling rocks into the paint can, this will depend on the amout of sample. Seal the lid on the paint can.

11.3.4 Secure the paint cans one on top of another with lids facing inward. Wrap duct tape (or equivalent) around to tightly secure.

11.3.5 Load paint cans onto ball mill, using the following procedure:

11.3.5.1 Start on the top set of rollers, adding paint cans from left to right.

11.3.5.2 Each ball mill has 3 sets of rollers. Each roller should be loaded in this order: 4 taped together samples, followed by 10 additional samples that are taped off in pairs (sets of 2, lids facing each other)

11.3.5.2.1 If there is an odd number of samples to roll, tape a can of sand to the last sample.

11.3.5.2.2 This method helps prevent sample loss by keeping the paint cans from rising off of the roller or the paint can lid from popping off

11.3.6 Start the ball mill.

11.3.7 Record the date and analyst's initials in the Dry/Grind Logbook to document when samples were put onto a ball mill.

11.3.8 The sample should tumble for at least 1 hour to provide adequate **mixing**; to ensure adequate **grinding**, the sample should roll for **8** hours.

11.3.9 At the end of the processing cycle, turn off the ball mill and remove the paint cans from the ball mill.

11.3.10 In a hood, open the paint cans and visually inspect the sample to ensure that the sample has been adequately ground. Once the sample is adequately ground, remove the rolling rocks.

11.3.11 Store in the appropriate location for laboratory analysis.

11.4 Rolling Rock Cleaning Process

NOTE: Prior to collecting rinsate for testing, send a request to the Technical Director or the QA department to create a job in LIMS. Include in your request how many containers are being tested, and the rinsate container ID's.

Samples will be logged in for 901.1_Cs/Fill_Geo_0 and 9310/Evaporation

11.4.1 Label the sides of 1 gallon paint cans as, "Dirty Rolling Rocks."

11.4.1.1 Add 46 rolling rocks and 40 oz of quartz sand per paint can.

11.4.1.2 Place one can on top of another, ensuring that the lids are facing inward. Wrap duct tape (or equivalent) around to secure cans to one another.

11.4.2 Load paint cans onto the ball mill and start rolling process.

11.4.2.1 Rolling rocks must roll for a minimum of 8 hours.

11.4.3 At the end of the processing cycle, turn off the ball mill and remove the paint cans.

11.4.4 In a hood, open the paint cans and transfer the contents to a 4-mesh sieve.

11.4.4.1 Pass the contents of a paint can through 4-mesh sieve to separate the sand from the rolling rocks.

11.4.4.2 Alternatively, the rolling rocks can be removed from the paint can and the sand brushed off by hand.

11.4.4.3 Frisk the sand to determine the appropriate waste stream for disposal.

11.4.5 Transfer rolling rocks to a bucket for radiac soaking.

11.4.5.1 Approximately 200 rolling rocks should be added per bucket.

11.4.5.2 Add enough radiac and water to cover the rolling rocks and achieve a 10-20% radiac solution (typically, 800 mL of the concentrated radiac is added per bucket)

11.4.5.3 Soak rolling rocks for a minimum of 4 hours.

11.4.6 After radiac wash, the rolling rocks need to be washed in a dishwasher.

11.4.6.1 Rinse thoroughly under faucet after dishwasher completion to remove any sanitizing chemicals that may remain.

11.4.7 Transfer rolling rocks to a 5 gallon cooler with spout for rinsate collection.

11.4.7.1 Place one bucket of cleaned rolling rocks into the container and fill with DI water until rolling rocks are submerged.

11.4.7.2 Collect a portion of the rinsate into an appropriately labeled 500 mL container and a 1 L Marinelli container. Add 2 mL of nitric acid to each container to acidify the sample, cap, and mix.

11.4.7.2.1 Record preservation information (pH, pH paper lot #, date and time of preservation, and HNO_3 lot#) in the Sample Preservation Logbook (Attachment 2)

11.4.7.2.2 Create a LIMS batch (method Fill_Geo_0) and record information. Submit the LIMS batch sheet along with the Marinelli to the count room.

11.4.7.3 Rinsate ID, Pass/Fail, Initials and Date must be recorded into Rolling Rock e-Log Book (Attachment 3)

11.4.7.3.1 Rinsates must meet criteria for being clean before rolling rocks may be used to grind/homogenize more samples.

11.5 Pulverizer procedure

11.5.1 Pulverize large solid samples such as rocks and cement samples, or samples which must be reduced to 200 mesh.

11.5.2 Fragment the sample to a size appropriate for the pulverizer dish.

11.5.3 Separate the larger particles that are not amenable to the pulverizer. Continue to fracture or fragment the larger portions until the entire sample can be added to the pulverizer.

11.5.4 In a hood, add sample to dish and puck.

11.5.4.1 **Do not overfill the dish and puck thereby preventing the lid from sealing securely.**

11.5.4.2 Record Puck ID and Date in the Puck ID Logbook (Attachment 4)

11.5.4.3 Record the Sample ID for each sample under the puck being used.

11.5.5 Place the dish securely in the pulverizer holder and close the door on the machine.

11.5.6 Open the air cylinder valve and regulate outgoing pressure to approximately 80 psi for operation of the pulverizer.

11.5.7 Press the green start button on the pulverizer.

11.5.8 To achieve 200 mesh sample size run pulverizer for five, 60 second intervals. Allow the assembly to cool between cycles as needed.

11.5.9 After pulverizing the sample, press the red stop button to stop the pulverizer.

11.5.10 Once the pulverizer has fully stopped (typically 10-15 seconds) lift the pulverizer door and remove the dish.

11.5.11 In a hood, carefully transfer the pulverized sample from the dish to a labeled secondary sample container.

11.5.11.1 Transfer the pulverized sample into a labeled paint can, add rolling rocks and mix for at least 1 hour on the ball mill.

11.6 Dish and Puck Cleaning Process

NOTE: Prior to collecting rinsate for testing, send a request to the Technical Director or the QA department to create a job in LIMS. Include in your request how many pucks are being tested, and the puck ID's.

Samples will be logged in for 901.1_Cs/Fill_Geo_0 and 9310/Evaporation

11.6.1.1 The dish and puck must be decontaminated before pulverizing the next soil sample to prevent cross contamination.

11.6.1.2 Vacuum the pulverizer and wipe with a damp paper towel as needed. Frisk the paper towel and dispose to appropriate waste stream.

11.6.1.3 Decontaminate the dish and puck by filling it with approximately 16 oz of quartz sand and replacing the lid. Then, place it in the pulverizer and operate the machine for 10 minutes.

11.6.1.4 Empty the sand. Brush off the dish, puck and lid. Use vacuum if necessary.

11.6.1.5 Rinse the dish, puck, and lid with radiac and wipe down with a paper towel. Then, rinse the dish, puck, and lid with DI water and wipe down with a paper towel. Wipe to ensure that all traces of sand are removed.

11.6.1.6 Wipe with methanol and let air dry.

11.6.1.7 **After pulverizing 20 samples in a dish, or after the last sample of a project has benn pulverized on a particular puck, whichever is less, a rinsate must be collected.**

11.6.1.7.1 Complete the dish and puck cleaning process.

11.6.1.7.2 Rinse lid, puck, and dish with approximately 1.5 L of DI water.

11.6.1.7.3 Collect a portion of the rinsate into an appropriately labeled 500 mL container and a 1 L Marinelli container. Add 2 mL of nitric acid to each container to acidify the sample, cap, and mix.

11.6.1.7.3.1 Record preservation information (pH, pH paper lot #, date and time of preservation, and HNO_3 lot#) in the Sample Preservation Logbook (Attachment 2)

11.6.1.7.3.2 Create a LIMS batch (method Fill_Geo_0) and record information. Submit the LIMS batch sheet along with the Marinelli to the count room.

11.6.1.7.4 Rinsates must meet criteria for being clean before puck may be used to pulverize more samples.

11.6.1.7.5 Rinsate ID, Analysis Run Date, Pass/Fail, and Initials need to be recorded in Puck Log Book (Attachment 4)

11.7 **Cone & Quartering**

11.7.1 Place a sheet of wax paper inside hood.

11.7.2 Pour sample from aluminum pan onto wax paper such that a cone is created. For samples with multiple pans, each additional pan should be poured onto the top of the cone so that the material is evenly and randomly distributed. Continue until the entire sample has been transferred.

11.7.3 Using a flat press, flatten the cone to approximately a uniform thickness, making sure that the sample remains well within the confines of the covered area.

11.7.4 Using a flat splitting tool, cut the sample into quarters by splitting once horizontally and once vertically.

11.7.4.1 Use the same tool to separate the quarters on the work surface.

11.7.5 Remove a quarter and transfer it back to the aluminum pan.

11.7.5.1 Weigh this portion. Sample weight needs to be 1600g ± 200g.

11.7.5.2 NOTE: When employing a "cone and quarter" technique for removing representative fractions of a sample, an entire quarter must be removed and, if necessary, may be further coned and quartered. **DO NOT** remove only a portion of a quartered sample, as this does not result in the removal of a representative sample fraction.

11.7.5.3 If sample weight exceedes the acceptable range:

11.7.5.3.1 Pour fraction from aluminum pan onto wax paper such that a cone is created. Flatten cone with press and quarter with splitting tool. Remove opposite quarters and continue in this manner until desired weight is obtained.

11.7.5.4 If sample weight is insufficient to meet the acceptable range:

11.7.5.4.1 Entire opposite quarter may be added or the opposite quarter may be coned and quartered until desire weight is obtained.

11.7.6 Transfer sample remaining on wax paper back into paint can and secure lid.

11.7.6.1 Print label containing Sample ID and place on the side of the paint can. Also label the can as "4 Mesh Archive".

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery, RPD, MDA) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 Include copies of the applicable pages of the pulverizer logbook with the data package.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification
- 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
  - 16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.2 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.3 Associated SOPs
- 17.3.1 ST-PM-0002, "Sample Receipt and Chain of Custody".

17.3.2 ST-QA-0002, "Standards and Reagent Preparation."
17.3.3 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
17.3.4 ST-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis."
17.3.5 ST-RD-0102, "Gamma Spectroscopy Analysis"
17.3.6 ST-RD-0403, "Gas Flow Proportional Counting Analysis"

18.0 CHANGES FROM PREVIOUS REVISION

18.1 Added section 11.3.6, requiring the recording of dates and analysts' initials in the oven temperature log to document when samples are put in the ball mill.

18.2 Revision 12:
- 18.2.1 Annual Review, No Changes.

18.3 Revision 13:
- 18.3.1 Added carbon-14 to section 4.0.
- 18.3.2 Updated the equipment in section 6.0.
- 18.3.3 Updated section 7.0 by adding a radiac and bleach wash solutions.
- 18.3.4 Updated the 9.1.2 and entire decontamination process in section 9.2.
- 18.3.5 Added instructions for frisking samples in section 11.2.
- 18.3.6 Added instructions for securing, taping and loading paint cans onto ball mill in section 11.3.
- 18.3.7 Updated section 13.2 regarding the decontamination of sample blanks.
- 18.3.8 Updated section 9.2.7 regarding transfering rolling rocks to a container for rinsate collection.
- 18.3.9 Added 11.3.2.1 requirement when to place a radioactive sticker on paint cans.
- 18.3.10 Updated section 11.4.12 on cleaning pulverizer and puck.
- 18.3.11 Removed decontamination blanks from section 13.0
- 18.3.12 Added attachment 1 and 2 to sop.
- 18.3.13 Replaced the use of quartz sand and replaced it with acidified water in section 9.0.
- 18.3.14 Updated labeling methods for Ball Mills in section 11.3.
- 18.3.15 Replaced the use of Methanol and replaced it with Ethanol in section 11.4.12.6.
- 18.3.16 Updated Section 15.

18.4 Revision 14: (07/31/2014)
- 18.4.1 grammatical corrections throughout SOP
- 18.4.2 added "For Radiochemistry Analysis" to SOP title
- 18.4.3 updated reference to Quality Manual as ST-QAM
- 18.4.4 updated Section 14 to indicate that QC information is maintianed in LIMS

18.5 Annual Review, No Changes (06/22/2015)

18.6 Annual Review, No Changes (05/23/2016)

18.7 Rev 15 (5/30/17)
- 18.7.1 Updated Section 5.2 to clarify ear protection requirements
- 18.7.2 Added Detail to Section 6: "Equipment"

18.8 Annual Review, No major changes (04/3/2018) Tech Review – M. Aldridge; QA Review – K. Ely
- 18.8.1 Updated signature page
- 18.8.2 Section 5: changed MSDS to SDS

18.9 Rev 16 (5/31/18) Tech Review – M. Aldridge; QA Review – K. Ely
- 18.9.1 Updated logbook names and attachments throughout.
- 18.9.2 Minor clarificaitons made throughout
- 18.9.3 Section 5: added Hazardous Material table
- 18.9.4 Section 6: updated equipment and supplies
- 18.9.5 Section 7:
  - 18.9.5.1 removed Ethanol, added Methanol
  - 18.9.5.2 added Nitric Acid
- 18.9.6 Section 9:
  - 18.9.6.1 Removed rolling rock cleaning process, moved this to section 11
  - 18.9.6.2 Added that rolling rock and puck mill cleaning requires testing by gamma and gross alpha beta.
  - 18.9.6.3 Added pass/fail criteria
  - 18.9.6.4 Formatted section 9.2 to look like section 9.1
- 18.9.7 Section 11

18.9.7.1 Updated to include options for large aliquot dry/grind throughout
18.9.7.2 Section 11.2: added a note warning about loss of volatiles
18.9.7.3 Section 11.4: added section about the rolling rock cleaning process
18.9.7.3.1 Added logging rinsates into TALS
18.9.7.3.2 Added recording preservation information
18.9.7.4 Section 11.5: clarified that pulverizer should be run for 5 – 60 second intervals
18.9.7.5 Section 11.6:
18.9.7.5.1 added logging rinsates into TALS
18.9.7.5.2 Added preservation of rinsate for gamma analysis
18.9.7.5.3 Added recording of preservation information
18.9.7.5.4 Section 11.7: New, Cone and Quartering

18.10 Rev 17 (03/22/19) Tech Review – M. Aldridge; QA Review – K. Ely
18.10.1 Minor clarifications made throughout
18.10.2 Section 6:
18.10.2.1 added additional container types
18.10.2.2 added mortar and pestle for grinding air filters
18.10.3 Section 11:
18.10.3.1 Included procedure for loading paint cans onto roller
18.10.3.2 Updated sections 11.4 and 11.6 to collect rinsate directly in 1L Marinelli instead of a 1L bottle.

18.11 Added Eurofins logo and updated copyright section (4/17/19)

Attachment 1

Location: Pre-Prep Oven Log

Logbook No.: 4614

TestAmerica
St. Louis

OVEN LOGBOOK

Lot ID	Oven#	Date In	Time In	Temp In °C	Accept Y	Accept N	Analyst	Date Out	Time Out	Temp Out °C	Accept Y	Accept N	Analyst	Ball Mill Date In

SOP References: ST-RC-0003
Form: QA-088, Rev.9

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Attachment 2

TestAmerica St. Louis

Sample Preservation Log

Logbook No: 4618

Sample ID	Initial pH	pH Paper Lot #	Date/Time of Preservation	HNO_3 Lot #	Final pH	Initials

INORG-0171 Rev 0 (05/11/2018)

1

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Attachment 3

Rolling Rock Cleaning

#	Rinsate ID*	Gamma Pass/Fail**	GAGB Pass/Fail**	Initials	Date	Batch/Comments
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						

Attachment 4

Logbook# 4543

		PULVERIZATION LOGBOOK									
Puck ID#											
Initials:											
DATE:											
Sample Number	1										
	2										
	3										
	4										
	5										
	6										
	7										
	8										
	9										
	10										
	11										
	12										
	13										
	14										
	15										
	16										
	17										
	18										
	19										
	20										
Job #											
Method		Gross A/B	Gamma	Gross A/B	Gamma	Gross A/B	Gamma	Gross A/B	Gamma	Gross A/B	Gamma
Analysis Date											
Pass/Fail*											
Initials											

*Do no reuse a puck until both tests have come back as "Pass". If either test comes back as "Fail", the puck must be re-cleaned and a new rinsate submitted

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INORG-0153, Updated 10/23/2018

Title: PREPARATION OF SAMPLES FOR GAMMA SPECTROSCOPY [EPA 901.1 and DOE GA-01-R]

Approvals (Signature/Date):

11/05/2019 Chelsea Mazariegos — Date Radiochemistry Prep Manager	11/6/19 Mike Ridenhower — Date Health & Safety Manager / Coordinator
11/6/19 Kristen Ely — Date Quality Assurance Manager	11/5/19 Sarah Bernsen — Date Radiochemistry Operations Manager

This SOP was previously identified as SOP No. ST-RC-0025 Rev. 18

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1.0 SCOPE AND APPLICATION

1.1 The purpose of this SOP is to provide detailed instructions for the preparation of samples which require gamma spectroscopy analysis.

1.2 This SOP describes methods for the preparation of samples of liquid, soil, vegetation, air filter, and core matrices prior to gamma spectroscopy analysis.

1.3 This SOP is based on EPA Method 901.1 and DOE Method GA-01-R.

1.4 The laboratory target analytes supported by this method, the reporting limits, and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Samples are transferred to a standard geometry container for counting on the gamma detectors. High purity germanium (HPGe) gamma detectors are used to detect isotopes with gamma ray energies between 40 and 2000 KeV. Activity concentration is determined using commercially available gamma spectral analysis software. A sample matrix which can be mounted in one of the standard geometries may be analyzed for any of the isotopes included in the radionuclide reference library. Detection limits may be affected by the sample size. Gamma photon energies not identified in the reference library may be identified and evaluated manually.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Replicate Analyses – Two or more analysis of the same sample whose independent measurements are used to determine the precision of the equipment's analytical procedure.

4.0 INTERFERENCES

4.1 Gamma energy emissions identified with scientifically measured probability by some radionuclides are documented by multiple sources. There are some discrepancies between reference sources and attempts are made to evaluate the reference data used in spectral analysis. Gamma emissions at discreet energy and probability are used to identify and quantify specific radionuclides in the sample. Gamma emissions which are completely absorbed by an HPGe detector form photo peaks which are used for identification and quantification of gamma emitting radionuclides. When two or more nuclides emit similar gamma energy the photo peaks cannot be resolved without using complex algorithms. These photo peaks in close proximity can interfere with the identification or quantification of a radionuclide. Knowing this, the nuclide reference library, computer software and analyst training are used to minimize the possibility of interference and misidentification. It is not possible to eliminate all interferences and misidentification.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all

samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 **Wear Kevlar or MAPA Blue-Grip gloves when using knives or sharp articles.**

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. ***NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.*** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm (TWA) 4 ppm (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Nitric acid can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1- Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance, top loader

6.2 Blender

6.3 Food chopper/grinder

6.4 Knives appropriate for food preparation

6.5 Graduated cylinder

6.6 Filter disk, 47 millimeter diameter

6.7 Plastic Tape

6.8 Marinelli beakers of various sizes (500 mL and 1L)

6.9 Petri dishes, 2-inch diameter

6.10 Can Sealer

6.11 Cans and lids (commonly referred to as tuna cans)

6.12 8 oz, straight sided polypropylene jars or equivalent; (used for 25 mL and 100 mL geometries)

6.13 Teflon® or glass beakers (250 mL, 400 mL)

6.14 Disposable digestion vessels

6.15 Muffle furnace (programmable)

6.16 TEXPEN®

6.17 Teflon® beaker covers

6.18 Watch glasses

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 DI Water

7.3 Nitric acid (16 M HNO_3) concentrated

7.3.1 Nitric acid (4 M HNO_3) – to an appropriately sized bottle containing 1500 mL of DI water; add 500 mL of 16 M HNO_3.

7.4 Hydrochloric acid (12 M HCL) – concentrated, 37.2%

7.5 Hydrofluoric acid (HF 48.52%) – concentrated

7.6 Radiacwash™ solution 10% - add 100 mL of Radiac to 1 L of water

7.7 Bleach solution 10% - add 100 mL of bleach to 1 L of water

7.8 Sodium Sulfate

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2, unless I-129 or I-131 is requested. Samples collected for I-129 or I-131 analysis are ***not preserved***.

8.3.1 The pH of aqueous samples is checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis

8.3.1.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

8.4 Milk samples are not chemically preserved.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process. Where no preparation method exists (i.e. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (DU).

9.2 **Method Blank (MB)**

9.2.1 The method blank is in the same geometry as the majority of the samples in the batch.

9.2.2 The Method Blank is a pre-prepared empty geometry that is analyzed with every batch.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 The LCS is in the same geometry as the majority of the samples in the batch.

9.3.2 An LCS is a blank matrix spiked with a known amount of analyte(s).

9.3.3 The LCS is either a purchased sealed source standard or is made in-house.

9.3.4 An LCS is in a pre-prepared container that is analyzed with every batch.

9.4 **Sample Duplicate (DU)**

9.4.1 A replicate analysis of the original sample counted on a different detector will be performed as the duplicate.

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6 **Decontamination of Tuna Can Sealer**

9.6.1 The sealer must be wiped down with 10% Radiacwash™ solution daily

9.6.1.1 Analyst must record the information in the daily logbook..

9.6.2 Sealer will be monitored for contamination as part of the monthly contamination survey, as per SOP ST-RP-0032

10.0 CALIBRATION AND STANDARDIZATION

10.1 The balance must be calibrated in accordance with ST-QA-0005.

10.2 For Gamma Spectroscopy calibration requirements, see ST-RD-0102.

11.0 PROCEDURE

11.1 See Attachment 2 for Automatic Canning Instructions

11.2 Liquid Sample Preparation

11.2.1 Liquid samples shall be prepared as a 25 mL, 100 mL, 500 mL, or 1L geometry.

11.2.2 Determine the proper geometry.

11.2.2.1 The volume of sample used depends on the amount required to meet the detection limits, the volume of sample supplied by the client, and whether the sample has very high activity. The sample volume may be reduced for high activity samples due to detector dead time considerations. Consult the count room supervisor or

radiochemistry technical director, if the sample has high activity which may require such consideration.

11.2.3 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.

11.2.4 Write sample information (i.e. ID #) on the geometry.

11.2.5 Measure the required sample volume (25, 100, 500 or 1L) by comparing the sample to the reference geometry.

11.2.5.1 Reference geometries are pre-made geometries comprised of DI water measured volumetrically.

11.2.5.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-specific geometry should be utilized (e.g. 500 mL or 1L Marinelli beaker) which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.2.5.3 If the client does not provide sufficient sample, and the sample is near a larger geometry, rather than reducing the volume significantly it may be preferable to dilute an aqueous sample with DI water to the correct volume in order to achieve a lower MDC. Consult Supervisor/Manager to determine which action is preferable.

11.2.5.3.1 If the sample is diluted, the undiluted volume is recorded as the sample volume. The dilution is only for fitting the calibrated geometry.

11.2.6 Place the lid securely on the geometry.

11.2.6.1 Remove excess air from Marinelli.

11.2.6.2 If the density is suspected to be greater than 1.2 g/ mL or less than 0.98 g/mL, generate a NCM. To determine the density use form RAD-0075_Density.xls (include form with batch paper work if utilized) Attachment 1

11.2.7 Seal the lid using plastic electrical tape. Marinelli beakers are prone to leaking liquids; the tape is tightly wrapped around the lid and the beaker in three layers each overlapping the previous layer with half the width of the tape. Make sure there are no creases in the tape which will form a channel for leakage.

11.2.8 Inspect for leakage.

11.2.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.3 Soil Sample Preparation

11.3.1 Soil samples for I-129 or I-131 analysis **are not dried and ground** but rather inserted into an appropriate calibrated geometry. Proceed to step 11.3.3.

11.3.2 Soil samples, which do not require I-129 or I-131 analyses, are prepared in accordance with SOP ST-RC-0003.

11.3.3 Soil samples shall be prepared as 230 mL sealed (tuna) can, 25 mL or 100mL straight sided poly jar, or 500 mL Marinelli geometry based on the amount of available sample. An Air Filter Geometry may be used if less than 25 mL of sample is available. In both the tuna can and marn soil geometries, the soil should nearly fill the geometry.

11.3.3.1 For I-129 analysis only a 25 mL or 100 mL straight sided poly jar geometry may be used (check with count room analyst on which geometry I-129 is calibrated for and prep the sample using the appropriate geometry).

11.3.3.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-

specific geometry should be utilized (e.g. tuna can or 500mL Marinelli beaker) which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.3.4 Write sample information (i.e. ID #) on the sample geometry.

11.3.5 Pre-weigh the empty geometry (tare weight) and record weight in TALS.

11.3.6 Fill the geometry with the appropriate amount of sample as described below.

11.3.6.1 Fill tuna cans above the ridge mark with sample. If there is insufficient sample, reduce geometry size.

11.3.6.2 Fill 100 mL geometry to the appropriate level by comparing to the reference geometry. If there is insufficient sample to fill, reduce the geometry size.

11.3.6.2.1 A 100 mL reference geometry is filled to the appropriate volume.

11.3.6.3 Fill 25 mL geometry to the appropriate level by comparing to the reference geometry. If there is insufficient sample, consult your supervisor or manager., If approval is given, an air filter geometry may be selected.

11.3.6.3.1 A 25 mL reference geometry is filled to the appropriate volume.

11.3.6.4 If air filter geometry is approved for use, spread soil evenly in a thin layer on the bottom of the petri dish. Do not put soil on a planchette; it needs to sit directly in the petri dish.

11.3.6.5 Fill 500 mL Marinelli beakers to the ridge mark just below the lid with sample. If there is insufficient sample to fill the marn soil to the ridge, reduce geometry size.

11.3.7 Close the sample geometry securely.

11.3.8 For tuna cans, seal with can sealer, wipe samples clean with paper towel and DI water.

11.3.9 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of geometry plus sample, minus the tare weight of the empty geometry).

11.3.10 For sampless that are not in tuna cans, seal the lid tightly using plastic electrical tape.

11.3.11 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.4 Vegetation Sample Preparation (No digestion)

11.4.1 Vegetation samples may be prepared in an appropriate calibrated geometry counted directly as dried and chopped matrix or green unprocessed matrix (if directed to do so by the client or if I-131 or I-129 is to be reported).

11.4.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by client or Project Manager.

11.4.1.1.1 Vegetation samples **<u>for I-129 or I-131 analysis are not dried</u>** but rather inserted into an appropriate calibrated geometry.

11.4.1.1.2 Dry weight can be determined on sample(s) not dried by using the percent moisture.

11.4.1.2 Consult the client requirements, client requirement memorandums or the Supervisor/Manager to determine proper sample handling.

11.4.2 The sample shall be counted in a 500 mL Marinelli, 230 mL sealed (tuna can), or a 25 mL or 100mL straight sided poly jar. The geometry is filled to the appropriate level with the sample.

11.4.2.1 I-129 analysis uses only a 25 mL or 100 mL straight sided poly jar geometry (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using the appropriate geometry).

11.4.2.2 Ra-226 is best reported inferred from the Bi-214 daughter after a 21-day ingrowth to allow the Ra-226 progeny through the potentially volatile Rn-222 daughter to reach secular equilibrium. To ensure Rn-222 is not lost from the geometry or does not escape into the headspace of the geometry, a radium-specific geometry should be utilized (e.g. tuna can or 500 mL Marinelli beaker)

which contains minimal headspace and can be sealed to prevent loss of radon. If Ra-226 is reported inferred from Bi-214 without sufficient ingrowth or from a geometry which is not intended for this purpose (due possibly to insufficient sample available), a NCM should be written to be included in the case narrative of the report.

11.4.3 Write sample information (i.e. ID #) on the geometry.
11.4.4 Pre-weigh the empty geometry and record weight in TALS.
11.4.5 Place sample in the tared geometry.
11.4.5.1 Compress the sample when filling a 500 mL Marinelli beaker
11.4.6 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of geometry plus sample minus the tare weight of the empty geometry).
11.4.6.1 Verify with the Project Manager if sample is to be reported on a wet or dry basis.
11.4.7 Close the sample geometry securely, seal with plastic electrical tape
11.4.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.5 Vegetation Sample Preparation (with digestion)
11.5.1 Vegetation samples may be digested and then placed into an appropriate calibrated geometry and counted as a liquid matrix.
11.5.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by the client or project manager.
11.5.1.1.1 **Iodine isotopes (e.g. I-125, I-129 or I-131) cannot be prepared using digestion technique** due to volatility.
11.5.1.2 Consult the client requirements and Supervisor/Manager to determine proper handling.
11.5.2 Consult your supervisor/manager if a vegetation sample requires digestion.
11.5.3 After sample digestion, label appropriate geometry and bring to volume to match the reference geometry.
11.5.4 Close the geometry securely and seal with plastic electrical tape.
11.5.5 Inspect for leakage.
11.5.6 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.6 Air Filters/Swipes (no digestion)
11.6.1 Air filters may be counted as single filters or as composite filters.
11.6.1.1 Consult client requirements and Supervisor/Manager for instruction.
11.6.1.2 Air filters are reported as pCi/sample or pCi/g. The weight of the air filter is required if the reporting units is pCi/g.
11.6.2 Write sample information (i.e. ID#) on Petri dish.
11.6.3 Filters with reporting units of:
11.6.3.1 pCi/g proceed to 11.6.4
11.6.3.2 pCi/sample proceed to 11.6.5
11.6.4 Pre-weigh empty Petri dish, record the weight in TALS and then on the lid of the Petri dish.
11.6.5 Load air filter(s) directly into Petri dish
11.6.6 Place lid on Petri dish, for pCi/g record the weight of the sample (geometry and sample weight, minus geometry weight) in TALS.
11.6.7 Secure the Petri dish lid with plastic electrical tape
11.6.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.7 Air Filters/Swipes (with digestion)
11.7.1 Air filters are digested and counted as a liquid.

11.7.2 Consult your supervisor/manager if a air filter or swipe sample requires digestion.After sample digestion. label appropriate geometry and bring to volume using reference geometry as a guide.
11.7.3 Place lid securely on the geometry.
11.7.4 Seal the lid using plastic electrical tape.
11.7.5 Inspect for leakage.
11.7.6 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.8 Core Samples
11.8.1 To obtain sample, cut Shelby tube or sample container into two pieces.
11.8.1.1 Using a rigid pipe cutter, cut the tube completely through.
11.8.1.2 Using a wire saw, cut through the sample.
11.8.1.3 Cuts should be made at 2 inch intervals.
11.8.1.4 Remove sample from every other sliced section of the Shelby tube.
11.8.1.5 Dry and grind the sample as described in SOP ST-RC-0003.
11.8.2 Aliquot 500 g of sample
11.8.2.1 If less than 500 g of sample is available, contact Supervisor/Manager for instruction.
11.8.2.2 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 mL, or 25 mL, or 500 mL Marinelli geometry based on the amount of available sample. In the tuna can geometry, the soil should nearly fill the geometry.
11.8.3 Pre-weigh the empty geometry and record the weight on the geometry lid and in TALS
11.8.4 Writer sample information (i.e. ID#) on the sample geometry
11.8.5 Place the dried sample into the geometry for counting.
11.8.6 Weigh and record sample weight in TALS.
11.8.7 Secure the lid on the geometry with plastic electrical tape.
11.8.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.9 Food: vegetables, produce, grain or animal feed:
11.9.1 Vegetables, produce and grain samples may be prepared in a 500 mL Marinelli beaker or 1 L Marinelli beaker geometry depending on the requested reporting limit and available sample volume. These matrices are counted directly as whole grain, chopped or blended produce or vegetable matrices **without drying** unless directed by the client to dry the matrix.
11.9.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.9.2 For vegetables and produce, prepare the sample by chopping with a knife on a cutting board or using a food processor.
11.9.3 Write sample information (i.e. ID #) on the appropriate geometry.
11.9.4 Pre-weigh the empty geometry and record weight on geometry and in TALS.
11.9.5 Place processed sample in the pre-weighed geometry.
11.9.6 Compress the sample when filling a 500 mL Marinelli beaker
11.9.7 Weigh sample and record the weight in TALS in grams.
11.9.8 Close the sample geometry securely, seal with plastic electrical tape.
11.9.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.10 Food: meat and fish:
11.10.1 Meat and fish may be prepared in tuna cans, an appropriately sized Marinelli beaker. or a 25 mL or 100 mL geometry depending on the requested reporting limit and available sample volume. These matrices are counted directly **without drying.**
11.10.1.1 Consult the client requirements and Supervisor/Manager for instruction.

11.10.2 For meat and edible portions of fish, prepare the sample by chopping with a knife on a cutting board.

11.10.2.1 Fish sample are to be filleted prior to chopping.

11.10.2.2 For analysis of fish **when the whole fish is required to be analyzed**, remove the head with a knife and cut the fish into pieces of appropriate size to easily fit into the Marinelli beaker without air voids. Place the heads in the middle portion of the Marinelli and surround it with pieces to eliminate air voids or spaces.

11.10.3 Write sample information (i.e. ID #) on the geometry.

11.10.4 Pre-weigh the empty geometry and record weight on geometry and in TALS.

11.10.5 Place processed sample in the pre-weighed geometry.

11.10.6 Compress the sample evacuating any space in the geometry when filling a Marinelli beaker

11.10.7 Weigh sample and record the weight in TALS in grams.

11.10.8 Close the sample geometry securely, seal with plastic electrical tape.

11.10.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.11 Store unused portions of sample in appropriately sized poly containers. Food and vegetation samples are to be refrigerated.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

12.3 Percent Moisture: Mass of original sample minus mass of dried sample divided by mass of dried sample.

12.3.1 $$p = \frac{W - D}{D}$$

p = fraction of total evaporable moisture content of sample
W = mass of the original sample
D = mass of dried sample

12.4 Density: Sample weight divided by the volume of said sample weight

12.4.1 $$d = \frac{m}{v}$$

d = density
m = mass
v = volume

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3 Training Qualification

14.3.1 The Supervisor/Manager has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in ST-QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented minimizing the potential for pollution to the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

16.2.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method EPA 901.1.

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, GA-01-R.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision.

17.5 Associated SOPs
- 17.5.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
- 17.5.2 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil and Grease Samples for Radiochemical Analysis
- 17.5.3 ST-RD-0102, GammaVision Analysis
- 17.5.4 ST-RP-0032, Instrumentation and Surveillance
- 17.5.5 ST-PM-0002, Sample Receipt and Chain of Custody
- 17.5.6 ST-QA-0002, Standard and Reagent Preparation
- 17.5.7 ST-QA-0036, Non-conformance Memorandum (NCM) process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 No Changes, Annual Review
19.2 Rev 11:
- 19.2.1 Inserted instructions regarding requirements for checking the pH of samples upon receipt and or prior to analysis in section 8.3.1.
- 19.2.2 Updated when sample ID's should be written on the container in section 11.0.
- 19.2.3 Inserted instructions for generating container ID labels throughout section 11.0.
- 19.2.4 Updated when weighing and recording sample weights/mass should be documented in gamma worksheet in section 11.0.
- 19.2.5 Added instructions for properly cleaning tuna cans before storage in section 11.2.9.

19.3 Rev 12:
- 19.3.1 Extensive revision to entire procedure
- 19.3.2 Addition of attachment 1

19.4 Rev 13:
- 19.4.1 Added sodium sulfate to section 7.0.
- 19.4.2 Updated section 9.2 regarding the matrix of method blanks.

19.5 Rev 14:
- 19.5.1 Updated vegetation sample with and without digestion throughout section 11.3 and 11.4.

19.6 Rev. 15: (8/30/2013)
- 19.6.1 Grammatical corrections and removal of references to QuantIMS through out
- 19.6.2 Section 3, added definition of replicate analyses
- 19.6.3 Section 8, removal of 180 day holding time
- 19.6.4 Section 9, explained that replicate will be used as duplicate for this procedure
- 19.6.5 Section 9.6, added "record in daily logbook"
- 19.6.6 Deleted record tare weight on lid in section 11.1.2
- 19.6.7 Deleted record weight of sample (container and sample weight minus container weight) on lid in section 11.1.8 , 11.2.9, 11.3.6
- 19.6.8 Added record weight in TALS throughout SOP
- 19.6.9 Added print proper paperwork throughout SOP

19.7 Rev. 16: (01/16/2015)
- 19.7.1 Grammatical corrections through out
- 19.7.2 Updated section 11.0
- 19.7.3 Added SOP references to Section 17: ST-RC-0014, St-RP-0032

19.8 Annual Review – No Changes (01/26/2015)
19.9 Annual Review- minor, non-procedural updates made (12/20/2016)
19.10 Revision 17 (12/15/17 Tech Review M. Minier/T Romanko; QA Review M Ward)
- 19.10.1 Added information pertaining to prep for Radium-226 in Sections 11.1, 11.2 and 11.3

19.11 Revision 18 (12/3/2018) Tech Review – M. Aldridge; QA Review – K. Ely
- 19.11.1 Made minor corrections throughout
- 19.11.2 Removed references to SOP ST-RC-0014 (SOP was archived)
- 19.11.3 Change references from normality "N" to molarity "M"
- 19.11.4 Section 9:
  - 19.11.4.1 updated section to match current practice
  - 19.11.4.2 removed duplicate paragraph about NCM's
- 19.11.5 Section 11:
  - 19.11.5.1 Added reference to Attachment 2
  - 19.11.5.2 Removed the requirement to record weight on container lid (section 11.2.5 and 11.4.4)
  - 19.11.5.3 Updated/clarified geometries used
- 19.11.6 Added Attachment 2 – Automatic Canning Machine Instructions

19.12 Added Eurofins logo, updated copyright information (4/17/2019)
19.13 Revision 19 (11/12/2019) Tech Review- C. Mazariegos; QA Review- K. Ely
- 19.13.1 Grammatical corrections throughout
- 19.13.2 Removed procedures for performing digestions and replaced with instructions to consult a supervisor from sections 11.5 and 11.7

19.13.3 Updated verbiage regarding containers to geometries
19.13.4 Clarified 11.3.6.2 and 11.3.6.3 procedure to fill to reference geometry level, and added supervisor approval needed for geometry smaller than 25 mL d=1
19.13.5 Added 11.3.6.4 air filter geometry procedure instructions for soil samples
19.13.6 Updated section 9.2 to reflect current practice using an empty geometry for all matrices as the method blank
19.13.7 Updated SOP names in section 17
19.13.8 Updated Attachment 1

Attachment 1

Eurofins TestAmerica St. Louis

DENSITY CORRECTION

Digi Tube Lot #: _____

Date: _____ Balance ID: _____

Analyst: _____ Pipette ID: _____

	Sample ID:	Container Wt (g)	Sample Volume (mL)	Sample + Container Wt (g)	Density (g/mL)	Original Sample Aliquot (g)	Adjust Sample Aliquot (mL)
1							
2							
3							
4							
5							
6							

Instructions:
Record the weight of a Class A digi tube in "Container Wt(g)"; record the volume of the sample added to the digit tube in "Sample Volume (mL)"; record the sample and digi tube in "Sample + Container Wt(g)." "Original Sample Aliquot" is the full sample size used for the actual analysis.

This density is to be used for sample aliquot correction when a sample is suspected of density less than or greater than 1. Include this document with batch paperwork.

Review is to be completed by a peer (someone other than the analyst handling the samples).

Reviewed by: _____ Date: _____

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Attachment 2

Automatic Canning Machine Instructions

OVERVIEW

1. These instructions apply to the "All American Manual Can Sealer" Model 225.
2. The machine seals a tuna can by operation of the crank. One arm (marked F for 'First') crimps the lid onto the can, and then the other arm (marked S for 'Second') shapes it correctly.
3. Currently a cordless drill is used instead of a manual crank handle.

OPERATION

1. Make sure the F arm will act first by turning the crank a little. If necessary, rotate the crank until the F arm is about to close on the can but has not yet done so.
2. Take the can with the lid on it, and set it on the round platform. Hold the lid down so you can guide the can to meet the round metal plate on top that holds down the lid, while turning the lever to raise the platform. This prevents misalignment.
3. Turn the crank shaft by operating the cordless drill. While pressing its switch with the right hand, hold its battery with the left hand to prevent the drill itself from rotating.
4. The F arm will crimp the lid, and then the S arm will shape the crimp. Continue rotating the crank until the S arm has finished and moved away from the can completely. Stop the drill.
5. Turn the lever to lower the platform. Remove the can.
6. If the crank shaft is turned in reverse, the platform rotates in reverse, lowering the can (often without the lid attached) and throwing the lever unexpectedly. This is undesirable.
7. The drill may come loose during operation. Tighten the keyless chuck regularly to prevent a loose connection and deformation of the crank shaft. When tightening take care to not deform the crank shaft.

MAINTENANCE

1. The following should be performed once a day during heavy usage, or once a week during light usage:
   A. There are 3 holes marked "OIL" along the top, on the left side. Put one drop of machine oil in each hole.
   B. There is 1 unmarked hole on the left side directly under the adjustment bracket and above the return spring for that arm. Put 3 drops of machine oil in that hole.
   C. The rotors on the chucks (marked F and S) each have a bolt, with a slot for a screwdriver. If you were to take out the bolt you would find that the slot continues down one side of the bolt, so that you may put a drop of oil on the slot and it will seep down into the raceway and lubricate the rotor. Put 1 drop of machine oil in each slot. Rotate to spread the oil.
   D. The roller on the adjustment bracket can use 1 drop of machine oil directly on the exposed portion, and/or 1 drop in the slot adjacent to its axis pin. Rotate to spread the oil.

2. The following should be performed every six months:
   A. Remove the gearbox cover on the left side by removing the left arm return spring, unscrewing the 5 cover bolts with a 5/32" hex wrench, then rotating the cover back and forth while pulling on it. Apply thick grease (the size of a gumball) on the top of the worm gear with a popsicle stick, and rotate the crankshaft to spread the grease all around the main gear. Repeat two more times. Also apply a fair amount of grease on the round axle anchor on the cover plate.
   B. Remove the gearbox cover on the front (marked "Automatic Master-Sealer") by inserting a thin object (i.e. a paperclip) into the hole and pulling it out. Apply a fair amount of grease all over the conical gear teeth with a popsicle stick. Reach all the way back on both sides. Put the cover back in carefully since it is very malleable tin. Do not hammer or hit. The sides may be flared out slightly if it is too loose.
   C. Turn the lever to raise the platform. Apply a thin film of grease on the twisting part at the bottom.

Attachment 2 (Continued)

D. Remove the rotors from the chucks and clean them, and the bolts, with solvent. Apply a thin film of grease with your finger on all parts that rub together including the bolt. Reassemble.

3. When cleaning the exterior of the machine with solvent, be careful not to allow the solvent to get into areas that should be lubricated. The solvent will quickly break down the oil or grease, leaving no lubrication.

CONFIGURATION

1. Each ARM ASSEMBLY consists of:
   A. A primary arm that holds a chuck (marked F or S) and its rotor.
   B. A secondary arm, marked CY-24, with an adjustment screw and nut.
   C. An adjustment bracket, marked CY-22, with a roller.

2. The chucks (marked F or S) may be swapped between arms, since the parts are symmetrical. Whichever arm holds the F is to be used first. Whether that is on the left arm or right arm does not matter, as long as the operator uses the F arm first. Currently the F is on the right arm.
3. The rotors on the F and S chucks are shaped differently, since they perform different functions. They are not interchangeable between chucks. Each one must use its own type of rotor (crimping vs. shaping). The rotor bolts are sized differently.
4. The adjustment screw and nut on the secondary arms (marked CY-24) affects the crimp (for the F arm) and the shaping of the crimp (for the S arm). See the ADJUSTMENT section below.
5. The adjustment bracket has several holes marked 1, 2, 3, 2-1/2, and S. These are can sizes and do not need to be changed since only one size of can is used, the #2 which it is currently set to.

ADJUSTMENT

1. Loosen the nut on the adjustment screw on each arm. Back out the screw 5-7 turns so that the rollers will not contact the lid when the arms are in the closed position.
2. Put a can with lid on the platform. Raise the can into position.
3. Rotate the crank until the F arm closes completely, although the roller should not be touching the lid.
4. Tighten the adjustment screw until the roller touches the lid. Continue until the slot of the screw is vertical.
5. Write down "0" on a piece of paper. This will keep track of the position of the screw.
   A. Tighten the adjustment screw 1/4 turn and write down "1/4" on the paper.
   B. Rotate the crank until the F arm has finished crimping the can, moves away, and the S arm starts to move.
   C. The S arm should not touch the can, since the adjustment screw is backed all the way out. Continue rotating the crank to allow the S arm to complete its cycle.
   D. Inspect the crimp. If it looks correct then continue; otherwise put the can back and repeat steps A-C above.
   E. If you went too far, resulting in an unsatisfactory crimp, back off the adjustment screw 1/2 turn and try again with a new can.

6. Repeat the same steps for the S arm. This shaping process will require a can that has been already crimped, so that is why the F arm was adjusted first. It is advisable to crimp it once and remove the can between S arm adjustments to avoid deformation from repetition.
7. When both arms have been adjusted, hold the adjustment screw in position with a screwdriver while tightening the nut with a wrench. Do not over tighten the nut.
8. Default settings:
   A. F arm, 3/4 turn from first contact with a fresh lid.
   B. S arm, 1/2 turn from first contact with a crimped lid.

Title: THE DETERMINATION OF TRITIUM IN WATER (AND OTHER FLUIDS), SOIL, SILICA GELS, AND GRAPHITE

Approvals (Signature/Date):

02/05/2019	9/9/19
Chelsea Mazariegos — Date	Michael Ridenhower — Date
Radiochemistry Prep Manager	Health & Safety Manager / Coordinator
9/9/19	9/4/19
Kristen Ely — Date	Sarah Bernsen — Date
Quality Assurance Manager	Radiochemistry Operations Manager

This SOP was previously identified as SOP No. ST-RC-0030 Rev. 21

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the procedure for the preparation of samples for the determination of tritium in water (and other fluids), soil (and other solids), and silica gels to determine inorganic tritium ("HTO"). It also includes an option for chemical oxidation of a limited sample aliquot to determine "total" tritium (including organically bound tritium, "OBT").

1.2 This procedure is based on EPA Method 906.0 and DOE Method H3-04-RC. The chemical oxidation presented is based upon EERF Method C-01, with the addition of persulfate to improve oxidation.

1.3 The instrument analysis is described in SOP ST-RD-0302.

1.4 Steam Distillation is applicable to silica gels, soils (water added) and ground, surface, drinking or other types of water samples, where sufficient volume exits to perform steam distillation. The sample is free from quenching agents that would prevent the use of the Liquid Scintillation Counter (LSC) generated quench curve for efficiency determination.

1.5 Direct Method is applicable to those liquids where distillation is impractical, yet the LSC generated quench curve efficiency would still be appropriate (body fluids, oils, oil-water mixtures). **Direct Counting is NOT acceptable for Drinking Water samples.**

1.6 Standard Addition is applicable to either of the two previous methods, when excessive quenching prevents a reasonable determination of counting efficiency. By manipulation of counting times, this procedure is appropriate for high activity or environmental samples. **This procedure is not acceptable for Drinking Water samples.**

1.7 Chemical oxidation is applicable to aliquots of water, soil, or other samples with a limited amount of organic matter. Furnace oxidation of samples is recommended for larger aliquots of sample or samples with larger organic matter content.

1.8 The reporting limits, method detectable activities (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP provides detailed instructions for the preparation of samples for the determination of tritium in water (and other fluids), soil and silica gels.

- 2.1.1 Steam Distillation - an aliquot of unpreserved water is steam distilled, and the condensate collected. Scintillation cocktail is added and following a suitable period of dark adaptation, the sample is counted in the LSC. Using a predetermined quench curve, a counting efficiency is assigned, and results are calculated.
- 2.1.2 Direct Method - a small volume of sample is transferred to a counting vial, cocktail is added, and the sample is counted following dark adaptation. A counting efficiency is assigned and results are calculated. **This is not used for Drinking Water samples.**
- 2.1.3 Standard Addition - a sample observed to exhibit excessive quenching by previous counting is spiked with a known amount of tritium. The sample is recounted, and the counting efficiency factor is accurately determined. This factor is then applied to the original counting data and results are calculated. **This is not used for Drinking Water samples.**
- 2.1.4 Chemical Oxidation – organic matter present in the sample matrix is oxidized to carbon dioxide and water by addition of permanganate, persulfate, and sulfuric acid. The oxidized sample solution is then steam distilled and the condensate collected.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 LSV – Liquid Scintillation Vial

4.0 INTERFERENCES

4.1 Tritium existing in background water is an interference. Slightly elevated levels of tritium are present in surface waters, therefore it is preferable to use water from a deep well source.

4.2 Most interferences can be overcome using one of the three methods described in section two. Distillation would remove most interferences from the sample.

4.3 The use of plastic vials may cause buildup of static charge and give erratic results. The instruments contain anti-static controllers but the additional preventative measure of wiping the vial with an anti static cloth aids in prevention of erratic static buildup. Wiping the plastic vials with an anti static dryer sheet works well to prevent the buildup and discharge of static.

4.4 Glass Liquid Scintillation Vials may contain low amounts of potassium which includes the beta emitting natural isotope, K-40. When glass LSC vials are used, they should be made from low potassium glass.

4.5 Nitrogen will generate chemical quench, samples should not be preserved with nitric acid.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
- 5.2.1 None.

5.3 PRIMARY MATERIALS USED
- 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.
- 5.3.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially

hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Material [1]	Hazards	Exposure Limit [2]	Signs and symptoms of exposure
Sodium Hydroxide	Corrosive Poison	2 ppm (TWA)	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Potassium Permanganate	Oxidizer	5 Mg/M^3 (Mn Compounds) (Ceiling)	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 mg/m^3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Nitromethane	Flammable Explosive	100 ppm (TWA)	Eye, skin, respiratory tract irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time weighted average			
STEL – Short term exposure limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Steam distillation setup, with heating mantles, flasks, condensers and Barrett trap

6.2 Liquid scintillation vials (LSV) - glass or plastic, as appropriate, 20 mL volume

6.3 Nalgene bottles or equivalent, 25 mL volume with screw cap

6.4 Centrifuge tubes, poly 50 mL with cap.

6.5 Calibrated pipettes - Eppendorf or equivalent

6.6 Adjustable dispenser for liquid scintillation cocktail, range; 1 to 20 mL

6.7 Boiling Chips

6.8 Analytical Balance

6.9 250 mL, 500 mL, and 1 L flask, round and flat bottom

6.10 20 mL Barett trap

6.11 Silica sand

6.12 Anti-static "dryer" sheets

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water, obtained from the Milli-Q unit.

7.3 Background water (dead water) – deionized/reagent water with a tritium content at or below ambient environmental level, (maximum of 400 pCi/L). Deep well water is preferable when it is determined to have a lower tritium concentration than the deionized/reagent water when lower client MDC requirements suggest the use of lower count-rate background water. The tritium of this water should be well characterized.

7.3.1 Raw dead water from unpurified sources (e.g. unprocessed well water) is distilled on the tritium wall to create background water. Once the distilled background water has been collected, it needs to be verified. Alternatively, deionized water from the laboratory DI system may be utilized if it meets client MDC requirements. Purchased background water may not need to be distilled if it is already processed. Create a batch in TALS/RadCapture and prepare 4 vials. 1 vial will be comprised of 10 mL of your current background water. The other 3 vials will be comprised of 10 mL of the new background water. Add 10 mL of Ultima Gold LLT to each sample. Shake the vials and wipe them down with a dryer sheet prior to turning them into the count room.

7.3.2 Once the background water passes verification (< 400 pCi/L), then a reagent label will be created in TALS. The verification paperwork will also need to be attached to the reagent document tab in TALS.

7.4 Tritium Solution, standard 100 – 400 dpm

7.5 Sodium Hydroxide pellets

7.5.1 10 N Sodium Hydroxide - Dissolve 100 g sodium hydroxide in 200 mL deionized water. Cool to room temperature and dilute to 250 mL with DI water.

7.6 0.5 M Potassium Permanganate - Dissolve 39.5 g potassium permanganate in 200 mL deionized water. Dilute with deionized water up to 500 mL.

7.7 Liquid scintillation cocktail - Packard Ultima Gold LLT (Low Level Tritium) or use the stated scintillation solution or its equivalent.

7.7.1 Each new lot of cocktail needs to be verified to ensure there are no interfences.
7.7.1.1 Prepare ICV as detailed in SOP ST-RD-0302 vials and submit to count room with proper paperwork. Apply acceptance criteria from ST-RD-0302 (± 10% of the known value).

7.8 Methanol

7.9 Potassium Permanganate 5%, (Non-critical concentration)
7.9.1 Add 5 g of potassium permanganate to 100 mL of DI water. Mix.

7.10 Potassium persulfate 5% w/v, LC20080-2 LabChem Inc, or potassium persulfate (crystalline powder). (Non-critical concentration)
7.10.1 Add 5 g of potassium persulfate to 100 mL of DI water. Mix.

7.11 Sulfuric Acid, 10%
7.11.1 Non-critical concentration
7.11.2 Dilute approximately 100 mL of concentrated Sulfuric Acid to approximately 500 mL of DI water. Mix.

7.12 Oxidizing Cocktail (Non-critical concentration)
7.12.1 Mix equal parts 5% potassium persulfate, 5% potassium permanganate, and 10% sulfuric acid.
7.12.2 Prepare fresh daily.

7.13 Nitromethane.

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples should not be preserved.

8.2.1 The pH will be verified prior to analysis,

8.3 The sample should be collected in a glass container.

9.0 QUALITY CONTROL

9.1 **Batch**
9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same reagent lots. Where no preparation method exists (e.g. water sample for volatile organics, water samples for anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, reagent lots and personnel.
9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), a Matrix Spike (MS) and Sample Duplicate (DU). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets.

9.1.4 Samples having different QC criteria, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across differing QC requirements provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC criteria.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water.

9.2.4 For Soil analyses, the method blank is comprised of sand.

9.2.5 For Oil analyses, the method blank is comprised of vaccuum pump oil.

9.2.6 The method blank must be analyzed on a different condensor per batch. The condensor needs to be randomly selected prior to analysis and documented in logbook.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Tritium.

9.3.4 For Soil analyses, the LCS is comprised of sand fortified with Tritium.

9.3.5 For Oil analyses, the LCS is comprised of vaccuum pump oil fortified with Tritium.

9.3.6 The LCS must be analyzed on a different condensor per batch. The condensor needs to be randomly selected prior to analysis and documented in logbook.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.4.4 MS Sample for silica gels requires a post-distillation spike.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and the utilization of a LCSD to demonstrate precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 A background sample is prepared and submitted for counting with each batch. The counts determined by the background sample are then subtracted from all samples counted in the batch including the blank.
10.1.1 A background sample consists of 10 mL "dead water" and 10 mL of ultima gold LLT.

10.2 Balance calibration must be checked each day of use. Refer to SOP ST-QA-0005 for details.

10.3 Pipette calibration must be checked each day of use. Refer to SOP ST-QA-0005 for details.

10.4 See analytical SOP for instrument calibration. ST-RD-0302

11.0 PROCEDURE

11.1 Steam Distillation (Water)
11.1.1 Rinse condensor lines with 5 mL of methanol and allow to dry completely (10-15 minutes)
11.1.2 Ensure that the sample container is capped tightly and shake well.
11.1.3 Initiate distillation paperwork.
11.1.4 Check that the pH ≥ 6.
11.1.4.1 If the pH is not ≥ 6, document the pH and generate an NCM.
11.1.4.2 Adjust the pH to ≥ 6 using 10.0 M NaOH (sodium hydroxide).
11.1.5 Transfer a 100 mL aliquot of sample to a round bottom flask.
11.1.6 Add approximately 6 sodium hydroxide pellets and 1 mL of 0.5 N potassium permanganate. (If a reduced aliquot is used, ex: 75 mL, then adjust the potassium permangantate accordingly, Ex: 0.75 mL)
11.1.7 Add boiling chips to prevent bumping in the distillation flask.
11.1.8 Attach the flask to the distillation setup and distill. For samples having a limited volume, 6 sodium hydroxide pellets and 0.1 g potassium permanganate crystals **may** be added instead of prepared solutions.
11.1.8.1 A Blank and an LCS consisting of a few boiling chips and 100 mL of DI water are prepared in the same manner as the samples. The LCS is spiked with a known amount of tritium (0.5 mL)
11.1.8.2 A matrix spike and sample duplicate should also be prepared.
11.1.9 Attach the flask to the condensor and Barrett trap and begin distillation.
11.1.10 Collect the distillate in a Barrett Trap, discarding the first 10 mL and the last 5 mL.
11.1.11 Ensure that the flasks do not boil dry.
11.1.12 Collect at least 50 mL of distillate in a clean labeled 50-mL centrifuge tube.
11.1.13 Turn off the heating mantles and allow the samples to cool to room temperature.
11.1.14 Discard any remaining residue to base waste.
11.1.15 Record the sample number on the cap of the liquid scintillation vial (LSV).
11.1.16 Collected sample must be at room temperature before an aliquot is taken.
11.1.17 Transfer 10 mL of distillate to a labeled LSV, using a pipet.
11.1.18 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.1.19 Cap the vial and shake it to ensure complete mixing.
11.1.20 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.1.21 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.1.22 Submit the samples to the counting lab for analysis.

11.2 Silica Gels
11.2.1 Rinse condensor line with 5 mL of methanol and allow to dry completely (10-15 minutes).
11.2.2 Tare a 1 L round bottom flask, the sample should fill the inside bottom half of the flask.
11.2.3 Transfer the sample to the tared flask and record the sample weight.

11.2.4 Blank and LCS consist of 30 g of boiling chips and 40 mL of DI water. The LCS is spiked with a known amount of tritium (0.5 mL).
11.2.5 Attach the flask to the distillation setup and distill the silica gels until the sample no longer produces water (approximately 6 hours). QC samples are distilled until at least 20 mL are collected. **Discard the first 5 mL of distillate.**
11.2.6 Collect all the water distilled into a clean labled 50 mL centrifuge tube.
11.2.7 Turn off the heating mantles and allow samples to cool to room temperature.
11.2.8 Weigh the sample to determine loss on drying. Record weight.
11.2.9 Return the dried silica gel to its original container.
11.2.10 Record the sample number on the cap of the LSV.
11.2.11 Transfer 10 mL of distillate to a liquid scintillation vial (LSV), using a pipet.
11.2.12 For the Matrix Spike, perform a post distillation spike on the distillate by removing 0.5 mL of distillate and adding a known amount of tritium (0.5 mL).
11.2.13 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.2.14 Cap the vial and shake it to ensure complete mixing.
11.2.15 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.2.16 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.2.17 Submit the samples to the counting lab for analysis.

11.3 Solids (soil, filters/swipes if not direct counts)
11.3.1 Rinse condensor line with 5 mL of methanol and allow to dry completely (10-15 minutes).
11.3.2 Tare a 120 mL plastic specimen cup and add 30 g of soil to the cup.
11.3.3 Add 40 mL of DI water to each sample.
11.3.4 Prepare a Blank and LCS using 40 mL DI water and 30 g washed silica sand. LCS is spiked with a known amount of tritium (0.5 mL).
11.3.5 Leach samples for at least 4 hours.
11.3.6 Transfer samples to 50 mL centrifuge tubes and centrifuge at 2000 rpm for at least 5 minutes.
11.3.6.1 If supernate is clear proceed to step 11.3.7.
11.3.6.2 If supernate is not clear filter using a 0.45 micron filter and then proceed to 11.3.7.
11.3.7 Transfer supernate to a 250 mL round bottom flask.
11.3.8 Attach the flask to the distillation setup and distill the samples until at least 20 mL are collected. QC samples are distilled until at least 20 mL are collected. **Discard the first 5 mL of distillate.**
11.3.9 Collect the water distilled into a clean labled 50 mL centrifuge tube.
11.3.10 Turn off the heating mantles and allow samples to cool.
11.3.11 Discard any remaining residue to base waste.
11.3.12 Record the sample number on the cap of the LSV.
11.3.13 Transfer 10 mL of distillate to a liquid scintillation vial (LSV), using a pipet.
11.3.14 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.
11.3.15 Cap the vial and shake it to ensure complete mixing.
11.3.16 Inspect each LSV to ensure that it is free of dirt, grease, etc.
11.3.17 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.
11.3.18 Submit the samples to the counting lab for analysis.

11.4 Direct Method (**used for Oils and at client request for other matrices. Can NOT be used for Drinking Water**)
11.4.1 Record the sample number on the cap of the LSV.
11.4.2 Ensure that the sample is capped tightly and mix well.
11.4.3 Transfer an aliquot to an LSV.
11.4.3.1 Aliquot = 10 mL for water based samples

11.4.3.2 Aliquot = 1-2 mL for oil sample, depending on the color or transparency of the sample. A density test will have to be performed to get an accurate weight for the oil.

11.4.4 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.

11.4.4.1 For oils add 18-19 mL of cocktail (Insta Gel Plus) to equal 20 mL in the vial. Shake.

11.4.5 Cap the vial and shake it to ensure complete mixing.

11.4.6 Inspect each LSV to ensure that it is free of dirt, grease, etc.

11.4.7 Using an anti static "dryer" sheet, wipe the side of the LSV to reduce static.

11.4.8 Record the sample number on the cap of the LSV.

11.4.9 Submit the samples to the cout room for analysis.

11.5 Internal Standardization (**Can NOT be used for Drinking Water)**

11.5.1 To the previously counted sample LSV, carefully open and add 1.0 mL of tritium SRM solution.

11.5.2 Replace the cap, shake well and wipe the LSV clean with a Kimwipe.

11.5.3 Record all data on the tritium data sheet.

11.5.4 Prepare one spiked sample, one blank, and a duplicate.

11.5.5 Submit the samples to the counting lab for analysis.

11.6 Graphite Samples

11.6.1 Using a cheese grater, grate enough of the sample to meet detection limits.

11.6.2 Leach for 12 hours in 40 mL of 20% KOH in a 120 mL plastic specimen cup.

11.6.3 Prepare a Blank and LCS by adding 100 mL of DI water to a 250 mL flask.

11.6.4 Spike the Matrix Spike and LCS with 0.5 mL of tritium.

11.6.5 Filter sample using 0.45 micron filter.

11.6.6 Add leachate to 250 mL round bottom flask and dilute to 100 mL with DI water.

11.6.7 Attach to distillation setup and distill.

11.6.8 Discard the first 5 mL of distillate to designated graphite waste container.

11.6.9 Collect at least 20 mL of distillate.

11.6.10 Allow distillate to cool to room temperature.

11.6.11 Aliquot 10 mL into a liquid scintillation vial.

11.6.12 Add 10 mL of Ultima Gold LLT or equivalent cocktail and shake thoroughly.

11.6.13 Inspect each LSV to ensure that it is free of dirt, grease, ect. Using an anti static "dryer" sheet, wipe the sides of the LSV to reduce static.

11.6.14 Submit samples to count room for analysis.

11.7 Chemical Oxidation

11.7.1 Rinse condensor lines with 5 mL of methanol and allow to dry completely (10-15 minutes)

11.7.2 If a water sample, ensure that the sample container is capped tightly and shake well.

11.7.3 Initiate distillation paperwork.

11.7.4 If the sample is water, check that the pH $\geq$ 6.

11.7.4.1 If the pH is not $\geq$ 6, document the pH and generate an NCM.

11.7.4.2 Adjust the pH to $\geq$ 6 using 10.0 M NaOH (sodium hydroxide).

11.7.5 Transfer the desired amount of sample (e.g. 75 mL aliquot of water sample or 1 g of soil/solid) to a round bottom flask.

11.7.6 Carefully add oxidizing cocktail to the flask. Samples containing significant carbonates may bubble vigorously. The amount of oxidizng cocktail needed will depend on the amount of sample used.

11.7.6.1 For 75 mL or 1 g of sample, use 25 mL of oxidizing cocktail.

11.7.6.2 For larger soil sample (or solid matrix other than soil) aliquots, consult with your Supervisor or Technical Director.

11.7.7 Attach the flask to the distillation setup without the Barett trap attached.

11.7.8 Turn the heating mantle on and bring the sample to a light boil for approximately 30 minutes.

11.7.8.1 The heat setting may need to be turned down partway through to keep the sample form boiling too vigorously.

11.7.9 Attach the Barett trap and increase the heat setting (if necessary) such that condensate begins to be collected in the Barett trap.

11.7.10 **Discard the first 5 mL of distillate collected,** and then continue distillation until at least 20 mL are collected. QC samples are distilled until at least 20 mL are collected. Do not collect more volume to help ensure oxidant is not brought over with the distillate.

11.7.10.1 If the collected 20 mL of sample condensate appears to be slightly discolored due to oxidant, or if it is suspected that oxidant has come over with the distillate, distill the collected condensate a second time using a fresh flask/distillation setup with nothing added except the condensate. Collect all condenstate during this second distillation, but don't distill to dryness.

11.7.11 Collect the water distilled into a clean labled 50 mL centrifuge tube.

11.7.12 Turn off the heating mantles and allow samples to cool.

11.7.13 Discard any remaining residue to acid waste.

11.7.14 Record the sample number on the cap of the LSV.

11.7.15 Transfer 10 mL of distillate to a liquid scintillation vial (LSV), using a pipet.

11.7.16 Using an automatic dispenser, add 10 mL of scintillation cocktail to the LSV.

11.7.17 Cap the vial and shake it to ensure complete mixing.

11.7.18 Inspect each LSV to ensure that it is free of dirt, grease, etc.

11.7.19 Using an anti static "dryer" sheet , wipe the sides of the LSV to reduce static.

11.7.20 Submit the samples to the counting lab for analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTEMANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA Method 906.0 "Tritium in Drinking Water"

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual Method, H3-04-RC

17.3 "Radiochemical Determination of Carbon-14 in Aqueous Samples", Method C-01, Eastern Environmental Radiation Facility Radiochemistry Procedure Manual, EPA, 520/5-84-006 (1984)

17.4 TestAmerica St. Louis Quality Assurance Manual (ST-QAM)

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (ST-HS-0002).

17.6 Associated SOPs

17.6.1 ST-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."

17.6.2 ST-RC-5006, "Decontamination of Laboratory Glassware. Labware and Equipment"

17.6.3 ST-RD-0302, "Operation and Calibration of a Liquid Scintillation Counter".

17.6.4 ST-QA-0002, "Standards and Reagent Preparation."

17.6.5 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.6.6 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
17.6.7 ST-PM-0002, "Sample Receipt and Chain of Custoday."

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA Method 906.0 has been expanded to include soil and other matrix samples. A chemical oxidation technique has been added for clients interested in "total" tritium (including organically bound tritium, "OBT").

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Rev. 10:
- 19.1.1 Revised equipment and supplies needed in section 6.0.
- 19.1.2 Updated Standards and Reagents in section 7.0.
- 19.1.3 Updated Procedural methods in section 11.0.

19.2 Rev. 11:
- 19.2.1 No Changes, Annual Review.

19.3 Rev. 12:
- 19.3.1 Listed background water as dead water in section 7.0.
- 19.3.2 Replaced the use of boiling chips with sand throughout section 9.0.
- 19.3.3 Defined the cocktail for dead water in section 10.1.1.
- 19.3.4 Updated other kinds of solids used in section 11.3.

19.4 Rev. 13:
- 19.4.1 Updated the instructions for analyzing the method blank and the LCS in section 9.2 and 9.3.

19.5 Rev. 14:
- 19.5.1 Added the requirement for a post-distillation spike for silica gel treated samples in section 9.4.

19.6 Rev. 15:
- 19.6.1 Updated § 11.2.11 to clarify language on post distillation spike.
- 19.6.2 In section 7.0, updated instructions for verifiying cocktail mixtures used in analysis.
- 19.6.3 Updated instructions for verifiying sample pH before analysis in section 8.0.
- 19.6.4 Updated sections 9.2 and 9.3 to require rotation of condensors used for method blanks and LCS's.

19.7 Rev. 16:
- 19.7.1 Updated section 15.
- 19.7.2 Updated supplies in section 6.0.
- 19.7.3 Updated silica gel procedures and solids in section 11.2 and 11.3.
- 19.7.4 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
- 19.7.5 Removed references to 'Clouseau" and "Quantims", replaced with LIMS.

19.8 Rev. 17: (08/11/2014)
- 19.8.1 Section 6.9, added 500-mL round and flat bottom flask to equipment and supplies used.
- 19.8.2 Grammatical errors fixed throughout
- 19.8.3 Section 8, Removed holding time
- 19.8.4 updated section 7.3 to detail process of using dead water to create background water

19.9 Rev. 18: (06/22/2015)
- 19.9.1 Grammatical errors fixed through out SOP
- 19.9.2 Updated section 11.0:
  - 19.9.2.1 Updated spike amounts
  - 19.9.2.2 Updated potassium permanganate amounts

19.10 Annual Review, No Changes (05/23/2016)

19.11 Rev 19: (05/30/2017)
- 19.11.1 Chemical oxidation option added, including section 1, 2, 5, 7, and 11.
- 19.11.2 Clarified section 7.3 in regards to background water (raw vs processed).
- 19.11.3 Sections 17 and 18 updated to include information/references regarding chemical oxidation.

19.12 Rev. 20 (09/28/2017)
- 19.12.1 Updated section 11.0 –Discard the first 10 mL and the last 5 mL. Collect at least 50 mL of distillate
- 19.12.2 Section 5: Changed reference to Safety Data Sheets from MSDS to SDS

19.13 Rev. 21 (07/17/2018)
- 19.13.1 Technical Review: S. Bernsen/QA Review M Ward
- 19.13.2 Updated section 7.0- identified "non-critical" reagents
- 19.13.3 Updated section 11- added "approximately" 6 sodium hydroxide pellets

19.14 Rev 22 (09/23/2019) Technical Review – C. Mazariegos, QA Review – K. Ely
- 19.14.1 Added Eurofins logo and updated copyright information.
- 19.14.2 Section 9 - Corrected acronym for sample duplicated (changed from "SD" to "DU")
- 19.14.3 Section 11.3 - Correction of step reference numbering.

Title: AQUEOUS AND SOIL SAMPLE PREPARTION FOR STRONTIUM-89, STRONTIUM-90 AND TOTAL STRONTIUM

Approvals (Signature/Date):

Chelsea Mazariegos 03/05/2019 Chelsea Mazariegos — Date Radiochemistry Prep Supervisor	Michael Riderhower 3/5/19 Michael Riderhower — Date Health & Safety Manager / Coordinator
Kristen Ely 3/5/19 Kristen Ely — Date Quality Assurance Manager	Andrew Buettner 3-5-19 Andrew Buettner — Date Operations Manager

This SOP was previously identified as SOP No. ST-RC-0050 Rev. 17

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1.0 SCOPE AND APPLICATION

Facility Distribution No.: 0	**Distributed To:** See Electronic Distribution Sheet

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1.1 This SOP describes the process for sample preparation of Strontium-89, Strontium-90 and Total Strontium. This procedure is applicable to aqueous samples and may be used in conjunction with soil pre-preparation procedures to perform Strontium determinations in solid matrices.

1.2 This SOP is based on EPA Method 905 and DOE EML HASL 300 Method, Sr-03-RC, Sr-02..

1.3 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Strontium is precipitated from a liquid matrix sample as insoluble carbonate. For soils, the Strontium is transferred from the soil into a liquid matrix prior to precipitation. Interferences from calcium and other radionuclides are removed by one or more precipitations of the Strontium carrier as Strontium nitrate. Samples with high dissolved solids can be precipitated as sulfates from an acid solution, eliminating most of the calcium interference. DTPA is added to the samples precipitated as sulfates to further remove interferences. Barium and radium are removed as chromate. The yttrium-90 daughter of Strontium-90 is removed by a hydroxide precipitation step. The yttrium-90 daughter grows in again and is then separated with stable yttrium carrier as hydroxide and finally precipitated as oxalate and beta counted. Strontium-90 and Strontium Total is counted for beta particle activity by Gas Flow Proportional Counters (GFPC). The Strontium-89 concentration is determined by difference.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Samples which contain significant natural strontium will cause inaccurate chemical yield determinations. Analysis for native concentration (e.g. via ICP) will allow for correction of the yield.

4.2 In hard water, large quantities of calcium precipitate with the Strontium in the initial carbonate precipitation. Repeated washes with concentrated nitric acid and precipitation of strontium as sulfate minimizes this potential interference.

4.3 Radioactive barium and radium, if present, are effectively removed by precipitation as the chromate.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Company Confidential & Proprietary
[THIS IS A CONTROLLED DOCUMENT. WHEN PRINTED IT BECOMES UNCONTROLLED]

5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Ammonium Hydroxide	Poison Corrosive	50 ppm - (TWA) (NH_3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Fuming Nitric Acid	Poison Corrosive Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation symptoms include breathing difficulties, coughing, choking, and irritation of the nose, throat, and respiratory tract. Onset of symptoms may be delayed 4-30 hours. Ingestion of nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. Skin contact can cause redness, pain, and severe skin burns. Vapors are irritating to the eyes.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Oxalic Acid	Corrosive	1mg/m^3 (TWA) 2mg/m^3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
Sodium Chromate	Corrosive Oxidizer Cancer Causing	For chromic acid and chromates, as CrO_3 = 0.1 mg/m^3 (ceiling)	Inhalation symptoms may include sore throat, coughing, shortness of breath, and labored breathing. Skin contact may include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain and severe tissue burns.
Sodium Hydroxide	Corrosive Poison	2 mg/m^3 (TWA)	Nose irritation; pneumonitis; eye, skin burns; temporary loss of hair
Hydrochloric Acid	Poison Corrosive	5 ppm (ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

Ceiling – At no time should this exposure limit be exceeded.

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (min. 4 place)

6.2 Centrifuge and centrifuge tubes

6.3 Quartz crucibles

6.4 Glass and teflon beakers, various sizes, covers and watch glasses

6.5 Planchets

6.6 Spatulas

6.7 Hot Plate

6.8 Hot bath

6.9 pH paper, narrow range

6.10 Desiccator

6.11 Digestion Vessels

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Deionized Water (DI water)

7.3 Acetic Acid (17.4 M), glacial CH_3COOH.

7.4 Ammonium Hydroxide (15 M) - concentrated NH_4OH.

7.5 Barium carrier

7.5.1 Barium carrier (10 mg/mL, Ba) - In a 1 L graduated cylinder, dissolve 19 g of Barium Nitrate in 500 mL of deionized water. While stirring carefully, add 5 mL of concentrated Nitric Acid and dilute to a final volume of 1 L with deionized water.

7.6 DTPA 0.2 M, Diethylenetriaminepentaacetic Acid, Pentasodium Salt (40 % in H_2O)., Dilute 200 mL of DTPA solution to 1000 mL with DI water.

7.7 Hydrochloric acid (12 M HCl) - concentrated, 37.2%.

7.8 Metacresol Purple Indicator (MCP) (0.1%) - In a volumetric flask, dissolve 0.5g of m-cresolsulfonephthalein (MCP) in deionized water. Add 2 mL of 10M NaOH and dilute to a final volume of 500 mL with deionized water.

7.9 Nitric Acid (90%) – fuming.

7.10 Nitric Acid (16 N) (69 – 71%) - concentrated HNO_3.

7.11 Hydrofluoric Acid, concentrated (29 N HF)

7.12 Oxalic Acid (0.4 M) - In a 1-L graduated cylinder, dissolve 52.02 g of oxalic acid, ($H_2C_2O_4$), in deionized water. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.13 Sodium Carbonate ($Na_2CO_3 \cdot 10H_2O$), (1 M) - In a 1 L graduated cylinder, dissolve 286 g of sodium carbonate, in deionized water. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.14 Sodium Chromate (Na_2Cr0_4), (1.5 M) - In a 1 L beaker, dissolve 242.96 g of sodium chromate in deionized water. Add the solution to a graduated cylinder. Dilute to a final volume of 1 L with deionized water. Mix thoroughly.

7.15 Sodium Hydroxide (NaOH), (10 M) - In a 1 L beaker, SLOWLY and CAREFULLY dissolve 400 g of sodium hydroxide pellets, in approximately 500 mL of deionized water. When the solution has cooled to room temperature, quantitatively transfer the solution to a graduated cylinder. Dilute to a final volume of 1 L with deionized water.

7.16 Sodium Hydroxide (NaOH) (10N)

7.17 Sodium Sulfate, Na_2SO_4, crystals.

7.17.1 Sodium Sulfate (Na_2SO_4), (0.7 M) - In a 1 L graduated cylinder, dissolve 100 g of sodium sulfate in deionized water. Dilute to a final volume of 1 L with deionized water.

7.18 Strontium carrier

7.18.1 Strontium Solution (10 mg/mL, Sr) In a 1 L volumetric flask, dissolve 12.077 g of Strontium Nitrate in approximately 250 mL of deionized water. With stirring, add 5 mL concentrated Nitric Acid. Dilute to a final volume of 1 L with deionized water.

7.19 Strontium Carrier (standardized) 52.5 mg/mL

7.19.1 If the Strontium carrier is not already standardized, standardize the Strontium carrier using the following procedure:

7.19.1.1 Prepare 3 samples of the carrier solution. Perform the following steps on each vial.
7.19.1.2 Add 6 mL of 0.2M DTPA to a 50-mL centrifuge tube.
7.19.1.3 Add 1.00 mL Strontium carrier
7.19.1.4 Add 2 drops metacresol purple indicator.
7.19.1.5 Add 10M NaOH with swirling until a purple endpoint is achieved.
7.19.1.6 Add 10 mL of 0.7M sodium sulfate.
7.19.1.7 Add approximately 3 mL of glacial acetic acid with swirling until a yellow solution is achieved.
7.19.1.8 Heat the solution for 5 - 10 minutes in a hot water bath.
7.19.1.9 Cool for 5 - 10 minutes in an ice bath.
7.19.1.10 Centrifuge for approximately 10 minutes at a speed sufficient to cause the precipitate to form a pellet.
7.19.1.11 Decant and discard the supernate in the appropriate waste container.
7.19.1.12 To the precipitate, add 20 mL of DI water
7.19.1.13 Vortex well, Centrifuge and decant.
7.19.1.14 Add 20 mL of DI water.
7.19.1.15 Vortex well, Centrifuge and decant.
7.19.1.16 Add approximately 5 mL of deionized water, slurry the precipitate.
7.19.1.17 Pre-weigh a planchette and record the tare weight.
7.19.1.18 Transfer onto a preweighted planchette with a spatula.

7.19.1.19 Heat to dryness on a hotplate at medium heat.
7.19.1.20 Remove from heat and cool to room temperature in a dessicator.
7.19.1.21 Weigh sample to determine Strontium yield.
7.19.1.22 Repeat heating and cooling in the dessicator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.
7.19.1.23 Record gross and final weights in the Rad Standards Log. Assign a unique number to the solution.

7.20 Ytrium carrier (standardized)

7.20.1 If the Yttrium carrier is not already standardized, standardize the Yttrium carrier using the following procedure:

7.20.1.1 Prepare 3 samples of the carrier solution. Perform the following steps on each vial.
7.20.1.2 Add 5 mL deionized water to a 50 mL centrifuge tube.
7.20.1.3 Add 2 mL concentrated nitric acid.
7.20.1.4 Add 1 drop of MCP indicator (0.1%).
7.20.1.5 Add 1.0 mL of yttrium carrier (10 mg/mL, Y)
7.20.1.6 Add 5 mL of 0.4 M oxalic acid.
7.20.1.7 **CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.**
NOTE: The pH must be between 1.7 - 1.9 to assure a uniform 9·H_2O hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.
Heat the solution for approximately 5 –10 minutes in a hot water bath.
7.20.1.8 Cool the solution for approximately 5 – 10 minutes in an ice water bath.
7.20.1.9 Centrifuge.
7.20.1.10 Decant supernate into the appropriate waste container.
7.20.1.11 Add approximately 20 mL of DI water to the precipitate.
7.20.1.12 Vortex. and Centrifuge.
7.20.1.13 Decant supernate into the appropriate waste container.
7.20.1.14 Add approximately 20 mL of DI water to the precipitate.
7.20.1.15 Vortex and Centrifuge.
7.20.1.16 Decant supernate into the appropriate waste container .
7.20.1.17 Add approximately 5 mL of D.I. water, slurry the precipitate.
7.20.1.18 Pre-weigh a planchet and record the tare weight on the sample worksheet.
7.20.1.19 Transfer the slurry to the planchette.
7.20.1.20 Dry the planchette on a hotplate.
7.20.1.21 Remove from heat and cool to room temperature in a desiccator.
7.20.1.22 Weigh sample to determine yttrium yield.
7.20.1.23 Repeat heating and cooling in the dessciator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.
7.20.1.24 Record gross and final weights in the Rad Standards Log. Assign a unique number to the solution.

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 The pH of aqueous samples are checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis.

8.3.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior ro analysis.

8.4 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.5 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Liquid analyses, the method blank is comprised of 1 L DI water acidified with 2 mL nitric acid.

9.2.4 For Soil, filter, wipe and vegetation analyses, the method blank is comprised of 1.25M calcium nitrate.

9.2.5 For leached analyses, the method blank is comprised of the leaching acid.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Liquid analyses, the LCS is comprised of 1 L DI water acidified with 2ml nitric acid fortified with Strontium-90.

9.3.3.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium 90.

9.3.4 For Soil,filter, wipe and vegetation analyses, the LCS is comprised of 1.25M calcium nitrate fortified with Strontium-90.

9.3.4.1 For Strontium-90 and/or Total Strontium, the LCS is fortified with Strontium-90.

9.3.5 For leached analyses, the LCS comprised of the leaching acid fortified with Strontium-90.

9.4 **Matrix Spike/Matrix Spike Duplicate**

9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and automatic pipetter calibrations must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

10.2 See analytical SOP ST-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System", for calibration requirements.

11.0 PROCEDURE

11.1 Water Samples

11.1.1 Initiate sample preparation worksheet.

11.1.2 Ensure that the sample container is capped tightly and shake it thoroughly.

11.1.3 Transfer a 1 L sample aliquot to a beaker.

11.1.3.1 For client requesting a reporting limit less than 2 pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.

11.1.3.2 If less than 1 L of sample was provided by the client, write the NCM noting insufficient volume.

11.1.3.3 Upon visual inspection, if the aqueous sample is suspected to have a high density (> 1.2g/mL, e.g. a brine or waste) or a low density (> 0.98g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).

11.1.3.4 Create the MB and LCS using DI water.

11.1.3.5 Add concentrate nitric acid to the MB, LCS and any reduced aliquot samples, to ensure the samples are acidic.

11.1.3.6 Proceed to step 11.5.1

11.2 Soil Samples

11.2.1 For soil samples prepare sample per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."
11.2.2 Weigh 1 g into a 50 mL glass beaker labeled with a TEXPEN.
11.2.3 Prepare a Method Blank and LCS using 1.25M calcium nitrate. Add enough to cover the bottom of the beaker/muffling vessel.
11.2.4 Wet samples with enough DI to saturate the matrix.
11.2.5 Spike the LCS and MS/MSD (if applicable) , with 1.0 mL of Sr-90 spike (10-40 dpm for the targeted requested isotopes).
11.2.6 Add 2 mL of standardized strontium carrier to samples and QC.
11.2.7 Gently dry the samples on a hotplate so that it will not splatter.
11.2.8 Place in oven at 600° and allow to muffle for four hours. Allow to cool.
11.2.9 Add approximately 5 mL of concentrated hydrochloric acid to the beakers and cover with a watchglass. Allow the samples to reflux for approximately 20-30 minutes.
11.2.10 Using a cut transfer pipet, scrape the sample to loosen off the beaker and transfer the samples to properly labeled mod block tubes or teflon beakers with concentrated nitric acid. Try to keep the total amount of acid around 5 mL.
11.2.11 Add approximately 10 mL of hydrofluoric acid to each sample.
11.2.12 Digest in mod block or in teflon beakers on a hot plate at >110° until dry.
11.2.13 Carefully add 10 mL concnetrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.
11.2.14 Digest in mod block or in teflon beakers on a hot plate at >110° until dry.
11.2.15 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block for 30min, or on a hotplate for 10-15 minutes.
11.2.16 Transfer to 400 mL beakers, rinsing with 4M HNO_3.
11.2.17 Bring up to volume of 200 mL with DI H_2O.
11.2.18 Proceed to step 11.5.1

11.3 Vegetation samples
11.3.1 Weigh an aliquot of sample into a glass beaker (typical aliquot is 1 g, a reduced or increased aliquot may be used depending on the RL).
11.3.2 Create a Method blank and LCS using 1.25M calcium nitrate. Add enough to cover the bottom of the beaker/muffling vessel.
11.3.3 Spike the LCS and MS/MSD (if applicable), with 1.0 mL of Sr-90 spike (10-40 dpm for the targeted requested isotopes).
11.3.4 Add 2 mL of standardized strontium carrier to samples and QC.
11.3.5 Add approximately 5 mL of concentrated nitric acid to all beakers and gently cook to dryness on the hot plate. Make sure the samples do not splatter.
11.3.5.1 Additional concentrated nitric acid may be added, if the vegetation has not broken down after the first nitric cook down.
11.3.6 Cover samples with a ribbed watch glass and place samples into an oven at 600° and allow to muffle for a minimum of 4 hours.
11.3.7 Add approximately 5 mL of concentrated hydrochloric acid to the beakers and cover with a watchglass. Allow the samples to reflux for approximately 20-30 minutes.
11.3.8 Using a cut transfer pipet, scrape the sample to loosen off the beaker and transfer the samples to properly labeled mod block tubes or teflon beakers with concentrated nitric acid. Try to keep the total amount of acid around 5 mL.
11.3.9 Add approximately 10 mL of hydrofluoric acid to each sample.
11.3.10 Digest in mod block or in teflon beakers on a hot plate at >110° until dry.
11.3.11 Carefully add 10 mL concentrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.
11.3.12 Digest in mod block or in teflon beakers on a hot plate at >110° until dry.
11.3.13 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block for 30 min, or on a hotplate for 10-15 minutes.
11.3.14 Transfer to 400 mL beakers, rinsing with 4M HNO_3.
11.3.15 Bring up to volume of approximately 250 mL with DI water.

11.3.16 Proceed to step 11.5.1 (skipping step 11.5.4)

11.4 Filters and Wipes

11.4.1 Filters and wipes analyzed for strontium only refer to SOP: ST-RC-0004.

11.4.2 Filters and wipes being split between analysis:

11.4.2.1 Take the appropriate aliquot and place in a 400 mL beaker, dilute sample aliquot with DI water to approximately 250 mL.

11.4.2.2 Create QC in a 400 mL beaker using approximately 250 mL of DI water.

11.4.2.3 Proceed to step 11.5.1

11.4.2.3.1.1 If the samples have already had their appropriate standards added, proceed to step 11.5.1 but skip step 11.5.4.

11.5 Precipitation

11.5.1 Add enough MCP indicator, while stirring, to see the red point of the indicator. If the samples are yellow instead of red, check the pH and add more concentrated nitric acid to ensure the samples are acidic. For soils, proceed to step 11.5.3.

11.5.2 Add 2.0 mL of standardized strontium carrier to all samples and QC. Add 1.0 mL of Sr-90 spike to LCS and MS/MSD if applicable.

11.5.3 Add 1 mL of barium carrier (10 mg/mL).

11.5.4 While stirring with a transfer pipet, add 10 N sodium hydroxide (NaOH) to the purple point of the indicator.

11.5.5 Place on a hotplate at approximately 400-450 degees and heat to near boiling as determined by visual observation.

11.5.6 While stirring with a transfer pipet, SLOWLY, add 10 mL 1M Na_2CO_3 solution.

11.5.7 Heat the samples approximately 15 minutes, stirring occasionally. Turn off the heat. **NOTE: Sample(s) should become cloudy indicating the formation of a precipitate. If no precipitate is visible, continue to add sodium carbonate solution until precipitation is achieved. If no precipitate forms after 20 mL Na_2CO_3, inform manager/supervisor before continuing.**

11.5.8 Cover the samples and allow the precipitate to settle for at least 6 hours at room temperature.

11.6 Strontium and Barium Precipitation

11.6.1 Decant off the supernate and discard into the base waste container.

11.6.2 Transfer the precipitate to a labeled centrifuge tube, rinsing with DI water as needed to transfer.

11.6.3 Centrifuge for approximately 5 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.6.4 Discard the supernate into the base waste container.

11.6.5 Repeat the centrifuge steps (11.6.3 – 11.6.4) as necessary to collect the entire sample in a single centrifuge tube.

11.6.6 SLOWLY add 10 mL of concentrated nitric acid to the precipitate in the centrifuge tube. Carefully swirl the tube until the precipitate is fully dissolved, and the sample is finished reacting.

CAUTION: The next step must be performed in a fume hood that has been designated for strong oxidizing acids. Wearing a facesheild, CAREFULLY add the fuming nitric acid.

11.6.7 CAREFULLY and SLOWLY add 20 mL fuming nitric acid to the centrifuge tube.

11.6.8 **CAUTION: Visually confirm that the chemical reaction is completed before capping the tube. Gas may form which may cause the sample to splash.**

11.6.9 Cap centrifuge tube, vortex vertically and cool in an ice bath for 10 minutes with occasional swirling to precipitate the Strontium and Barium Nitrates.

11.6.9.1 If a precipitate does not form, add additional fuming nitric acid in 10 mL increments until precipitate forms. Chill in an ice bath until the precipitate is settled.

11.6.9.2 If after adding 20 mL of fuming nitric acid addition, a precipitate still has not formed, contact manager/supervisor for instruction.

11.6.10 Centrifuge for 5-10 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.6.11 Discard supernate to acid waste container.

11.6.12 Add 10 mL concentrated nitric acid, vortex vigorously to completely break up the precipitate.

11.6.13 Heat in hot water bath for approximately 5 minutes.

11.6.14 Centrifuge for 5-10 minutes at a speed sufficient to cause the precipitate to form the shape of a pellet.

11.6.15 Discard supernate in the acid waste container.

11.6.16 Repeat steps 11.6.12 to 11.6.15 until the mass of the sample appears similar to the mass of the method blank and LCS.

11.6.17 Dissolve the precipitate in 10 mL of deionized water.

11.6.18 Vortex.

11.6.18.1 If any insoluable solid remains, centrifuge for 5-10 minutes at a speed sufficient to shape any insoluble solids in the form of a pellet.

11.6.18.2 Transfer supernate to a unused labeled centrifuge tube. Set aside any insoluble residue.

11.7 Strontium Sulfate Precipitation

11.7.1 Add 2 drops MCP indicator.

11.7.2 Add 5 mL DTPA.

11.7.3 Dropwise, add 10N sodium hydroxide to the sample with swirling until you observe a purple color change.

11.7.3.1 Dropwise add 10N sodium hydroxide in excess, color will not change (typically 5 extra drops).

11.7.4 Add 5 mL of 1.5 M sodium chromate.

NOTE: It is important to add the glacial acetic acid of the next step slowly so that it does not prematurely precipitate the Strontium.

11.7.5 While mixing CAREFULLY and SLOWLY, add 17.4 M glacial acetic acid dropwise until the color of the solution changes from golden yellow to reddish-orange. The pH should optimally be 5.3. Check pH with narrow range pH paper, if the color does not resemble reddish orange and if not precipitate forms.

11.7.6 Heat for at least 5 minutes in a hot water bath.

11.7.7 Cool the solution for 5 - 10 minutes in an ice bath.

11.7.7.1 If a precipitate does not form, the solution may be "seeded" with 1-2 drops of Na_2CrO_4 mixed with 1-2 drops of Barium solution.

11.7.8 Centrifuge for approximately 5 minutes at a speed sufficient to form the precipitate into the shape of a pellet.

11.7.9 Transfer the supernate to a clean 50 mL unused, labeled centrifuge tube.

11.7.10 To the supernate add 10 mL of 0.7M sodium sulfate.

11.7.11 Add 7 mL 17.4M glacial acetic acid to precipitate the Strontium sulfate.

11.7.12 Record the current date and Time as "T1", the start date and time of Y-90 ingrowth, and TSR on the laboratory worksheet.

11.7.13 Centrifuge. Decant the supernate to the acid waste container.

11.7.14 Add approximately 10 mL of DI water, vortex to slurry the solution. Centrifuge. Decant off the supernate into the acid waste container.

11.7.14.1 If the precipitate is still yellow/orange, repeat DI water step, 11.7.14.

11.7.15 For Total Strontium (only) analyses proceed to 11.11.

11.8 Ingrowth

11.8.1 Dissolve the Strontium sulfate precipitate from Step 11.7.15 in 10 mL of 0.2 M DTPA.

11.8.2 Vortex thoroughly.

11.8.2.1 If precipitate is visually apparent, heat to complete dissolution.

11.8.3 Add Yttrium carrier to all samples

11.8.4 Add 1 drop MCP.

11.8.4.1 The color should be purple.

11.8.4.2 If the color is not purple, add 10N NaOH, dropwise, until a purple color is obtained.

11.8.4.2.1 If more than 1 mL NaOH has been added and the color is not obtained, contact manager/supervisor for instruction.

11.8.5 Cap the tube and store for 7 to 14 days to permit ingrowth of yttrium 90.

11.9 Out of In-growth

11.9.1 Add 5 mL of 0.7 M sodium sulfate.

11.9.2 **NOTE: From step 11.9.2 through step 11.10 are to be completed before the end of the working day. If all steps can not be completed within the prescribed time, do not start the next step. Contact manager/supervisor for instruction.**

11.9.3 Precipitate strontium sulfate by adding 3 mL 17.4M glacial acetic acid.

11.9.4 Record the current date and time, "T2", as the end date and time of the in-growth period of ^{90}Y on the sample worksheet.

11.9.5 Heat the solution for 5 - 10 minutes in a hot water bath.

11.9.6 Cool the solution for 5 - 10 minutes in an ice bath.

11.9.7 Centrifuge for approximately 5 minutes at a speed sufficient to cause the precipitate to form a pellet.

NOTE: In the next section of this procedure, ^{89}Sr, ^{90}Sr is separated from ^{90}Y to determine a separate activity for ^{90}Y, and for Strontium.

11.10 Yttrium Yield

11.10.1 Transfer the supernate to a unused, labeled centrifuge tube.

11.10.1.1 **Cap and save the precipitate fraction for step 11.11**

11.10.2 To the supernate, add 2 drops of MCP indicator.

11.10.3 While swirling, add 15 mL 10N sodium hydroxide to the sample to achieve the purple endpoint.

11.10.4 Heat the solution for 5 - 10 minutes in a hot water bath.

11.10.5 Cool the solution for 5 - 10 minutes in an ice water bath.

11.10.6 Centrifuge for approximately 10 minutes at a speed sufficient to cause the precipitate to form a pellet.

11.10.7 Decant, discarding the supernate into the base waste container.

11.10.8 Dissolve the precipitate in 2 mL of concentrated nitric acid.

11.10.9 Add 5 mL of DI water.

11.10.10 Add 5 mL of 0.4 M oxalic acid.

11.10.11 Add 1 drop of MCP indicator.

11.10.12 CAREFULLY, add concentrated ammonium hydroxide dropwise until a pink-orange end point is obtained. **NOTE: The pH should be between 1.6 - 1.9 to assure a uniform $9 \cdot H_2O$ hydrate yttrium oxalate precipitate.** If needed, verify the pH with narrow range pH paper.

11.10.12.1 If pH > 1.9, add concentrated nitric acid dropwise.

11.10.13 Heat the solution for approximately 5 minutes in a hot water bath.

11.10.14 Centrifuge for approximately 5 minutes. Decant supernate to acid waste.

11.10.15 Add approximately 10 mL of DI water to the sample.

11.10.16 Vortex to slurry the sample.

11.10.17 Centrifuge for approximately 5 minutes. Decant supernate to acid waste.

11.10.18 Plating

11.10.18.1 Pre-weigh planchet and record weight in TALS worksheet.

11.10.18.2 Add a minimal amount of DI water to the precipitate.

11.10.18.3 Slurry the precipitate.

11.10.18.4 Transfer sample slurry onto a pre-weighed planchette.
11.10.18.5 Dry the planchette on a hotplate. While the sample is drying, occassionally swirl the precipitate on the planchet to ensure the precipitate dries evenly.
11.10.18.6 Remove from heat and allow sample to cool to room temperature in a desiccator for a minimum of 20 minutes.
11.10.18.7 Re-weigh the planchet and record the weight in the TALS worksheet to determine the chemical yield of the yttrium carrier solution.

11.11 Strontium Yield
11.11.1 Add approximately 10 mL of DI water to the precipitate.
11.11.1.1 For Sr-89 and/or Sr-90, precipitate is from 11.10.1.1.
11.11.1.2 For Total Strontium, precipitate is from 11.7.15.
11.11.2 Vortex to slurry the sample.
11.11.3 Centrifuge for approximately 5 minutes. Decant supernate to acid waste.
11.11.4 Plating
11.11.4.1 Pre-weigh planchet and record weight in TALS worksheet.
11.11.4.2 Transfer the sample slurry to the pre-weighed planchet with a minimal amount of water.
11.11.4.3 Dry the planchet on a hotplate. While the sample is drying, occassionally swirl the precipitate on the planchet to ensure the precipitate dries evenly.
11.11.4.4 Remove from heat and allow sample to cool to room temperature in a desiccator for a minimum of 20 minutes.
11.11.4.5 Re-weigh the planchet and record the weight in the TALS worksheet to determine the chemical recovery for the Strontium carrier solution.

11.12 Submit the planchet for counting to determine the Strontium activity (Total strontium)
11.12.1 If not able to submit to the count room, hold planchet in desiccator until ready.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Density of aqueous samples: sample mass divided by the density equals the volume.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1. Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.

14.2. Demonstration of Capability
14.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3. Training Qualification

14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Sr-03-RC, Strontium -90 in Environmental Samples, Sr-02, Strontium-90.

17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs (Current Revisions)

17.5.1 ST-RC-0002, Preparation of Stainless Steel Planchets for Radiochemistry Analyses

17.5.2 ST-RC-0003, Drying and Grinding of Soil and solid Samples
17.5.3 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis
17.5.4 ST-RC-5006, Decontamination of Laboratory Glassware. Labware and Equipment
17.5.5 ST-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System
17.5.6 ST-QA-0002, Standards and Reagent Preparation
17.5.7 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.8 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.5.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Modifictions to method 905:
- 18.1.1 Fuming nitric, instead of concentrated nitric acid, is used to remove interferences.
- 18.1.2 DTPA is used during the barium chromate precipitation instead of ammonium acetate buffer in the EPA method. DTPA complexes the strontium and helps it to stay in solution.
- 18.1.3 MCP (meta-cresol purple) indicator is used throughout in the place of methyl red.
- 18.1.4 Strontium is precipitated as a sulfate salt rather than as a carbonate salt as in the EPA method.
- 18.1.5 The iron hydroxide scavange step in the EPA procedure is omitted; interferences are minimized through the various precipitation steps in our procedure.

18.2 Modifications to method Sr-03-RC:
- 18.2.1 The multiple fuming nitric acid precipitations in the DOE method are reduced to just one in this SOP.
- 18.2.2 DTPA is used during the barium chromate precipitation instead of a barium buffer solution. DTPA complexes the strontium and helps it to stay in solution.
- 18.2.3 Strontium carrier, not Strontium-85 tracer, is used to determine yield.
- 18.2.4 The iron hydroxide scavange step in the DOE method is omitted; interferences are minimized through the various precipitation steps in our procedure.
- 18.2.5 In place of the filtration steps in the DOE method, the samples are centrifuged.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes, Annual Review
19.2 Rev. 10,
- 19.2.1 Added Sr-02 and Stronium-90 to sections 1.0 and 17.0.

19.3 Rev. 11:
- 19.3.1 Added reference to section 18.2 regarding samples being centrifuged in place of filtered.
- 19.3.2 Updated section 6.0 by adding new equipment.
- 19.3.3 Removed strontium-89, NIST traceble from section 7.0.
- 19.3.4 Updated information regarding pH checking of samples acidified and not acidified by sample control in section 8.3.
- 19.3.5 Added instruction for leached analyses in section 9.2 and 9.3.
- 19.3.6 Updated procedure for high and low density samples in section 11.1.
- 19.3.7 Added calculation for determining the density of aqueous samples in section 12.0.

19.4 Rev. 12:
- 19.4.1 Grammatical corrections throughout
- 19.4.2 Removed 180 day holding time requirement from section 8
- 19.4.3 Updated section 15

19.5 Rev. 13: (07/31/2014)
- 19.5.1 Prcedure updated in section 11.0
- 19.5.2 Grammatical errors fixed through out SOP

19.6 Rev. 14: (08/11/2015)
- 19.6.1 Grammatical errors fixed through out SOP
- 19.6.2 Updated Section 11.0
- 19.6.3 Section 11.2.4 added teflon beakers
- 19.6.4 Section 11.2.7 added on a hotplate
- 19.6.5 Section 11.2.10 added on a hotplate for 10-15 minutes
- 19.6.6 Section 11.4.6 added carefully swirl tube
- 19.6.7 Section 11.5.12 moved when to record the T1 time
- 19.6.8 Annual Review, No Changes (08/12/2016)

19.7 Rev. 15 (8/23/2017)
- 19.7.1 Grammatical errors fixed through out SOP
- 19.7.2 Updated section 5.0- changed MSDS to SDS
- 19.7.3 Updated reagents in section 7.0
- 19.7.4 Updated section 11.0: added Sr-90 spike aliquot amount and changed 10M to 10N.

19.8 Rev. 16 (12/22/2017 Tech Review by T. Romanko/S. Bernsen; QA Review M.Ward)
- 19.8.1 Updated section 4.0- additional information on interferences.
- 19.8.2 Updated section 18 to inlcude differences with original method.
- 19.8.3 Updated Section 4 – added additional information on interferences
- 19.8.4 Updated Section 11
- 19.8.5 Added additional procedures for acidifying samples
- 19.8.6 Moved additiona of standards to later in the precipitation procedure
- 19.8.7 Clarified steps in the procedure
- 19.8.8 Added observation and instructions based on sample color
- 19.8.9 Added additional instructions for plating
- 19.8.10 Updated Section 18 detailing modifications to the original method

19.9 Revision 17 (11/20/2018)
- 19.9.1 Technical Review: S. Bernsen
- 19.9.2 Updated section 9.0- updated the QC matrix for soil, filter, wipe and vegetation.
- 19.9.3 Updated section 11.0
- 19.9.4 11.2- Added instructions for spiking and tracing samples prior to muffling.
- 19.9.5 11.3- Added procedure for vegetation samples.
- 19.9.6 11.4- Added procedure for filter and wipe samples.
- 19.9.7 Updated formatting in Section 19

19.10 Revision 18 (03/08/2019)
- 19.10.1 Technical Review: C. Mazariegos/QA M Ward
  - 19.10.1.1 Corrected grammar and spelling throughout.
  - 19.10.1.2 Updated step number references throughout.
  - 19.10.1.3 Section 11.5.1 added "for soils, proceed to step 11.5.3.".
  - 19.10.1.4 Section 11.5.2 moved spiking and tracing prior to adding reagents that could cause precipitation.

19.11 Added Eurofins logo and updated copyright information (4/19/2019)

Title: SAMPLE PREPARATION FOR STRONTIUM-89, STRONTIUM-90 AND TOTAL STRONTIUM USING EXTRACTION CHROMATOGRAPHY

Approvals (Signature/Date):

03/26/2021	03/29/2021
Chelsea Mazariegos — Date Department Manager	Michael Ridenhower — Date Health & Safety Manager / Coordinator
3/26/2021	03/29/2021
Kristen Ely — Date Quality Assurance Manager	Sarah Bernsen — Date Operations Manager

This SOP was previously identified as SOP No. ST-RC-0058 Rev. 6

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes the process for sample preparation of strontium-89, strontium-90 and total strontium using extraction chromatography. This procedure is applicable to water and solid matrices.

1.2 This SOP is based on ASTM Method C1507-07 and Eichrom Method SRW01..

1.3 The reporting limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Strontium is isolated/separated by extraction chromatography. For waters, the strontium is pre-concentrated with calcium as the phosphate, then brought back up into a liquid matrix. For soils, the strontium is transferred from the soil into a liquid matrix prior to loading on the extraction column/cartridge. Interferences from calcium and other radionuclides are effectively removed by the extraction column. The separated strontium is eluted off the column, evaporated to dryness onto a planchet, and weighed for chemical recovery determination. For reporting total strontium (or strontium-89), the planchet is beta counted soon after planchet preparation, For strontium-90 determination, the strontium nitrate with ingrown yttrium-90 is dissolved from the planchet and loaded back onto the extraction chromatography column. The yttrium-90 passes directly through the column, is evaporated to dryness on a planchet, and beta counted. Strontium-90 and total strontium are counted for beta particle activity by gas flow proportional counting (GFPC). The strontium-89 concentration is determined by difference.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Samples which contain natural strontium cause inaccurate chemical yield determinations. For samples suspected to contain significant elemental strontium, the concentration should be determined by suitable means, and the yield calculation appropriately corrected.

4.2 The extraction resin effectively removes most beta-emitting isotope interferences (e.g. Ba-140 and K-40) utilizing 8 M nitric acid load and rinse solutions. Certain tetravalent elements, if present, (e.g. uranium, plutonium, neptunium, cerium, and ruthenium) are effectively removed using a 3 M nitric acid – 0.05 M oxalic acid wash.

4.3 The extraction resin has limited capacity based upon the number of sites available to bind the strontium. The carrier should be limited to about 5 mg to avoid overloading the column, resulting in lowered chemical recoveries. Alternatively, cartridges could be stacked to increase the load capacity.

5.0 SAFETY

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5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, non-absorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Oxalic Acid	Corrosive	1 mg/m3 (TWA) 2 mg/m3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
Hydrochloric Acid	Poison Corrosive	5 ppm (ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (capable of an appropriate precision as required by each measurement within this procedure)

6.2 Centrifuge and centrifuge tubes

6.3 Labware, glass and teflon beakers, various sizes, covers and watch glasses

6.4 Planchets

6.5 Digestion vessels

6.6 Syringes

6.7 Mod block

6.8 Hot plate

6.9 Desiccator

6.10 Strontium-specific extraction chromatography resin – Eichrom Technologies, Inc® Sr resin 2-mL pack cartridge.

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Deionized Water (DI)

7.3 Ammonium hydrogen phosphate (3.2M) (non-critical reagent)
7.3.1 Dissolve 104 g of $(NH_4)_2HPO_4$ in approximately 200 mL of DI water, heat gently to dissolve, and dilute to approximately 250 mL with DI water.

7.4 Ammonium hydroxide (NH_4OH), Reagent.

7.5 Bromocresol Purple indicator solution (non-critical reagent)
7.5.1 Dissolve 0.2 g of Bromocresol Purple (520.24 F.W.) in approximately 250 mL of water, add 1 mL of concentrated Ammonium Hydroxide.

7.6 Calcium nitrate (1.25M) (non-critical reagent)
7.6.1 Dissolve 51 g of $Ca(NO_3)_2$ in approximately 100 mL of DI water and dilute to approximately 250 mL with DI water.

7.7 Hydrochloric acid (12 M HCl) - concentrated, 37.2%.

7.8 Nitric acid (16 N HNO_3) (69 – 71%) - concentrated HNO_3.

7.9 Nitric acid (8 M HNO_3)

7.9.1 Non critical concentration
7.9.2 In a marked 2L bottle, add approximately 1L of DI water. Then SLOWLY and CAREFULLY add approximately 1L of concentrated HNO3
7.9.3 Shake carefully to mix.

7.10 Nitric acid (4 M HNO_3)
7.10.1 Non critical concentration
7.10.2 In a marked 2.5L bottle, add approximately 1875 mL of DI water. Then SLOWLY and CAREFULLY add approximately 625 mL of concentrated HNO3.
7.10.3 Shake carefully to mix.

7.11 Nitric acid (0.05 M HNO_3)
- 7.11.1 Non critical concentration
- 7.11.2 Add approximately 800 mL of DI water to a 1L container. Add approximately 3mL of concentrated HNO3.
- 7.11.3 Dilute to approximately 1L with DI water.
- 7.11.4 Shake carefully to mix.

7.12 Nitric acid (3 M HNO_3)/oxalic acid (0.05 M $C_2H_2O_4$) solution
- 7.12.1 Non critical concentration
- 7.12.2 In a 1L container, disslove 6.3 g of oxalic acid dihydrate in approximately 700 mL of DI water.
- 7.12.3 Add approximately 190 mL of concentrated HNO3 with mixing.
- 7.12.4 Dilute to approximately 1 L with DI water.

7.13 Nitric acid (3 M HNO_3)/boric acid (0.25 M BH_3O_3) solution
- 7.13.1 Non critical concentration
- 7.13.2 In a 1L container, dissolve 15.5 g of boric acid in approximately 700 mL of DI water.
- 7.13.3 Add approximately 190 mL of concentrated HNO3 with mixing.
- 7.13.4 Dilute to approximately 1 L with DI water.

7.14 Hydrofluoric acid, concentrated (29 N HF)

- 7.14.1

7.15 Strontium carrier (standardized) 5 or 25 mg/mL, NIST traceable
- 7.15.1 If the strontium carrier is not already standardized, standardize the strontium carrier using the following procedure with 6 replications:
  - 7.15.1.1 Add 5 mg (or 0.2 mL of 25 mg/mL SrNO3 solution) to 10 mL of 8M HNO3.
  - 7.15.1.2 Follow 11.4 column separation and plating.
  - 7.15.1.3
  - 7.15.1.4 Record gross and final weights in the Rad Standards Log and allow a manager or QA to assess the data to verify the replications are acceptable. Assign a unique number to the solution.

7.16 Strontium-89, NIST traceable

7.17 Strontium-90, NIST traceable

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

9.0 QUALITY CONTROL

9.1 **Batch**
- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised

of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate (DU). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.4 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For **soil** analyses, the method blank is comprised of calcium nitrate.

9.2.4 For water analyses, the method blank is comprised of DI water acidified with nitric acid.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For **soil** analyses, the LCS is comprised of calcium nitrate fortified with strontium-90.

9.3.4 For water analyses, the LCS is comprised of DI water acidified with nitric acid and fortifide with strontium-90.

9.4 **Matrix Spike/Matrix Spike Duplicate**

9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and automatic pipetter calibrations must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Laboratory Support Equipment."

10.2 See analytical SOP ST-RD-0403, "Low Background Gas Flow Proportional Counting (GFPC) System", for instrument calibration requirements.

11.0 PROCEDURE

11.1 Water Samples

11.1.1 Initiate sample preparation worksheet.

11.1.2 Ensure that the sample container is capped tightly and shake it thoroughly.

11.1.3 Transfer sample aliquot to a beaker; generally 1L is used.

11.1.3.1 Upon visual inspection, if the aqueous sample is suspected to have a high density (> 1.2 g/mL, e.g. a brine or waste) or a low density (< 0.98 g/mL, e.g. mixed solvent), the sample density will be measured and the volume determined arithmetically (sample mass divided by the density equals the volume).

11.1.3.2 Create the MB and LCS using DI water and acidify to a pH <2 with nitric acid

11.1.4 Add 5 mg strontium equivalent of standardized strontium carrier (either 0.2 mL of 25 mg/mL carrier or 1 mL of 5 mg/mL carrier) to samples and QC. Add about 5x the client's requested limit of Sr-90 spike to the LCS and MS/MSD, if applicable.

11.1.5 Add 2 mL of 1.25M $Ca(NO_3)_2$ to each sample and QC.

11.1.6 Add 5mL of 3.2M $(HN_4)_2HPO_4$ solution per liter of sample.

11.1.7 Add enough bromocresol purple indicator, while stirring, to see the color of the indicator (should be yellow at this point). If the samples are purple instead of yellow, check the pH and add more concentrated nitric acid to ensure the sampels are acidic.

11.1.8 Place samples on a hot plate and begin heating to near boiling. After the sample has reached near boiling (should see steam from the sample), turn the heat down to about medium.

11.1.9 While stirring add enough NH_4OH to reach the purple indicator end point and form $Ca_3(PO_4)_2$ precipitate. Allow the sample to heat for another 20-30 minutes.

11.1.10 Remove from the hot plate, and allow the sample to cool and the precipitate to settle.

11.1.11 Decant the excess supernate to a base waste container.

11.1.12 Transfer the precipitate to a labeled centrifuge tube and centrifuge the preicipitate for approximately 5 minutes at 2000 rpm.

11.1.13 Decant the supernate and discard to base waste.

11.1.14 Wash the precipitate with 10 mL of DI water. Vortex or shake to ensure the precipitate is thoroughly washed.

11.1.15 Centrifuge for approximately 5 minutes at 2000 rpm.

11.1.16 Decant the supernate and discard to base waste.

11.1.17 Dissolve the precipitate in 10 mL of 8M nitric acid.

11.1.18 Proceed to column loading (11.3).

11.2 Soil Samples (aliquot of less than 2.5 g or non-soil solids such as resin beads)

11.2.1 For soil samples, prepare sample as per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."

11.2.2 Weigh approximately 1 g of sample into a labeled beaker. Larger aliquots (up to 10 g) may be utilized, but acid volumes need to be adjusted accordingly. See your supervisor or technical director for instructions.

11.2.3 Add sufficient calcium nitrate to cover the bottom of the method blank and LCS beakers.

11.2.4 Add a small amount of DI water to all soil samples.

11.2.5 Add 5 mg strontium equivalent of standardized strontium carrier (either 0.2 mL of 25 mg/mL carrier or 1 mL of 5 mg/mL carrier) to samples and QC. Add about 5x the client's requested limit of Sr-90 spike (usually 1 mL) to the LCS and MS/MSD, if applicable.

11.2.6 Gently dry the beakers on a hotplate.

11.2.7 Place in muffle oven at 600° and allow to muffle for 4 hours. Allow to cool.

11.2.8 Add 10 mL concentrated hydrochloric acid to the beakers and cover with a watchglass. Heat at a low temperature (approximately 200 degrees) for 20-30 minutes to reflux solids off the bottom of the beaker.

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11.2.9 Using a cut transfer pipette, scrape the sides and bottom of the inside of the beaker and rinse the solids with concentrated nitric acid (about 5 mL) into a teflon beaker or digestion tube, completely removing the sample.

11.2.10 Add 10 mL concentrated hydrofluoric acid to the teflon beakers or digestion tubes. Cook until samples are completely dry.

11.2.11 Add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid, and 10 mL concentrated hydrofluoric acid to the teflon beakers or digestion tubes, and cook until completely dry once more.

11.2.12 Add 10 mL 8M HNO3 to samples and cover with a watchglass, heating for about 15-20 minutes to dissolve the solids.

11.2.13 Transfer samples to 50 mL centrifuge tubes. Proceed to column separation section 11.4.

11.3 Soil Samples (aliquot of 2.5 g of soil or larger)

11.3.1 For soil samples, prepare sample as per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."

11.3.2 Weigh up to 2.5 g of sample into a labeled beaker. Larger aliquots (up to 10 g) may be utilized, but acid volumes need to be adjusted accordingly. See your supervisor or technical director for instructions.

11.3.3 Add sufficient calcium nitrate to cover the bottom of the method blank and LCSbeakers.

11.3.4 Add a small amount of DI water to all soil samples.

11.3.5 Add 5 mg strontium equivalent of standardized strontium carrier (either 0.2 mL of 25 mg/mL carrier or 1 mL of 5 mg/mL carrier) to samples and QC. Add about 5x the client's requested limit of Sr-90 spike to the LCS and MS/MSD, if applicable.

11.3.6 Gently dry the beakers on a hotplate.

11.3.7 Place in muffle oven at 600° and allow to muffle for 4 hours. Allow to cool.

11.3.8 Add 10 mL of concentrated hydrochloric acid to all crucibles and cover with a plastic watch glass. Reflux on a hotplate for 20-30 minutes at approximately 200-300 degrees.

11.3.9 Use a cut transfer pipet and scrape the inside of the beaker to remove all solids. Transfer to digestion tube using approximately 5 mL of concentrated nitric acid.

11.3.10 Add an additional 25 mL of concentrated nitric acid to each digestion tube.

11.3.11

11.3.12 Digest in mod block at > 110° for 1-2 hours

11.3.13 Allow the solution to cool.

11.3.14 Transfer the solution/solids to a labeled centrifuge tube with minimal DI water and centrifuge. Carefully transfer the supernatant to a labeled beaker.

11.3.15 Add 10 mL of concentrated nitric acid to the solids remaining in the centrifuge tube and vortex to loosen the solids. Transfer the solids/solution to the original digestion tube. Rinse the tube with 10 mL concentrated nitric acid to ensure all the soil transfers and add to the digestion tube. Add 10 mL more nitric acid and 10 mL concentrated hydrochloric acid to the digestion tube.

11.3.16 Digest in mod block at > 110° for 1-2 hours.

11.3.17 Allow the solution to cool.

11.3.18 Transfer the solution/solids to a labeled centrifuge tube with minimal DI water and centrifuge. Carefully transfer/combine the supernatant with the supernatant from the first digestion.

11.3.19 For samples which are not expected to contain refractory strontium, proceed to step 11.1.15. The following steps are to be used for samples expected to contain refractory strontium or for which the client requires total dissolution. The client should notify the lab if the sample(s) received may contain refractory strontium.

11.3.19.1 Add 10 mL of concentrated nitric acid to the solids remaning in the centrifuge tube and vortex to loosen the solids. Transfer the solids/solution to a labeled teflon beaker.

11.3.19.2 Add 30 mL of concentrated hydrofluoric acid.

11.3.19.3 Cover the beaker and digest for several hours at low heat.

11.3.19.4 Remove the cover and evaporate to dryness.
11.3.19.5 If necessary to dissolve remaining residue, add 10 mL of concentrated nitric acid and 30 mL of concentrated hydrofluoric acid and repeat the digestion/evaporation steps above.
11.3.19.6 Add 15 mL 3 M nitric acid/0.25 M boric acid and evaporate to dryness.
11.3.19.7 Add 10 mL 8 M nitric acid, cover, and heat just to boiling for 5 minutes.
11.3.19.8 Cool, and then transfer the solids/solution to a centrifuge tube with minimal DI water and centrifuge. Carefully transfer/combine the supernatant with the supernatant from the initial digestions.

11.3.20 Carefully evaporate the combined supernatant to dryness.
11.3.21 Add 15 mL of 4 M nitric acid to the beaker and reflux for 10-15 minutes. Do not allow the volume to drop below 5 mL.
11.3.22 Transfer the solution to a labeled mod block tube.
11.3.23 Rinse the beaker with 5 mL of 4 M nitric acid. Transfer to the labeled mod block tube.
11.3.24 Carefully reduce the volume to 10 mL. If the volume drops below 10 mL, but greater than 5 mL, bring up to 10 mL with DI water. If the volume drops below 5 mL, consult your supervisor or technical director for instruction.
11.3.25 Proceed to column loading (11.4)

11.4 Initial Column Loading/Eluting

11.4.1 Prepare a labeled 2 mL extraction cartridge with a reservior (e.g. syringe barrel) and waste container and condition with 5 mL of 8 M nitric acid on a vacuum box setup.
11.4.2 Transfer the 10 mL sample to the reservior and allow to load at 1 mL per minute (approximately 1 drop every 3 seconds).
11.4.3 Rinse the cartridge with 5 mL of 8 M nitric acid at approximately 3 mL per minute (approximately 1 drop every second).
11.4.4 Rinse the cartridge with 5 mL of 3 M nitric acid/0.05 M oxalic acid solution at approximately 3 mL per minute.
11.4.5 Rinse the cartridge with 5 mL of 8 M nitric acid at approximately 3 mL per minute.
11.4.5.1 **Note: Record in LIMS, the date/time of this step as the final strontium rinse, indicating the beginning of the yttrium-90 ingrowth ("T1").**
11.4.6 Remove the acid waste container (discard to acid waste) and replace with a labeled collection tube.
11.4.7 Elute the strontium from the column with 10 mL of 0.05 M nitric acid at 1 mL per minute.
11.4.8 Evaporate the strontium eluant onto a cleaned/pre-weighed planchet (tare weight should be recorded in LIMS) by adding small portions (3-5 mL) to the plancheton a hot plate and allowing each portion to evaporate to near dryness between additions.
11.4.9 Evaporate completely to dryness, cool in a dessicator, and re-weigh. Record the final gross weight in LIMS.The expected strontium mass added (based on the amount added as a standardized carrier) is used to calculate strontium carrier recovery gravimetrically.
11.4.10 For total strontium or strontium-89 analysis, submit the prepared planchet(s) to the count room for analysis. Store in a dessicator until count is performed.
11.4.11 Save the labeled extraction cartridge and planchet if strontium-90 analysis is to be performed.

11.5 Yttrium-90 preparation

11.5.1 For strontium-90 analysis, hold the planchet and allow the yttrium-90 to ingrow for 7-14 days.
11.5.2 Condition the extraction cartridge from the initial strontium separation.
11.5.2.1 Prepare the labeled extraction cartridge saved from the initial strontium separation (or a new cartridge) with a reservoir (e.g. syringe barrel) and acid waste container on a vacuum box.
11.5.2.2 For a *new cartridge*, proceed to 11.4.2.4.

11.5.2.3 For a *reused cartridge*, rinse with 5 mL of 0.05 M nitric acid at 3 mL per minute. Note: the 0.05 M nitric acid removes Bi-210 that might be present due to Pb-210 from the sample tightly bound to the resin.

11.5.2.4 Condition the cartridge with 5 mL of 8 M nitric acid at 3 mL per minute.

11.5.2.5 Remove the acid waste container (discard acid waste) and replace with a labeled collection tube.

11.5.3 Dissolve the strontium nitrate residue from the planchet with up to 3 portions of 5 mL of warm 8 M nitric acid and store in a tube to be loaded onto the cartridge.

11.5.4 Load the 8 M nitric acid solution containing the dissolved residue onto the prepared cartridge at 1mL per minute a collect the rinse that falls through the resin for plating.

11.5.5 Rinse each cartridge with an additional 5 mL of 8 M nitric acid. Collect this rinse with the rinse from step 11.4.4.

11.5.6 Record in LIMS, the time of the last rinse as the stop time for the yttrium-90 ingrowth ("**T2**").

11.5.7 Evaporate the yttrium eluant (combined from steps 11.4.3 and 11.4.4) onto a cleaned/pre-weighed planchet (tare weight should be recorded in LIMS) by adding small portions (3-5 mL) to the planchet on a hot plate and allowing each portion to evaporate to near dryness between additions.

11.5.8 Evaporate completely to dryness, cool in a dessicator, and reweigh. Record the final gross weight in LIMS, to be used in the efficiency determination. The yttrium yield is assumed to be 100%.

11.5.9 Submit the prepared planchet(s) to the count room for analysis. Store in a dessicator until count is performed.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. precent recovery and realtive precent difference "RPD") and standard instrument software calculations are given in the TestAmerica St. Louis QAM (ST-QAM). Specific analysis calculations are given in the applicable analysis SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1. The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

14.2. Demonstration of Capability

14.2.1. Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3. Training Qualification

14.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM

15.0 VALIDATION

15.1 This method is based upon a published ASTM procedure which presents performance results for over 30 measurements made over 8 years using 16 different samples in duplicate from the Department of Energy – Environmental Measurements Laboratory – Quality Assurance Program. The laboratory has also validated this method using a MARLAP Level C type protocol for total strontium.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 ASTM Method C1507-07, "Standard Test Method for Radiochemical Determination of Strontium-90 in Soil". Current edition approved June 1, 2007.

17.2 Eichrom Technologies, Inc., Analytical Procedure SRW01 ("Strontium-89/90 in Water"), February 2003.

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs (Current Revisions)

17.5.1 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis

17.5.2 ST-RC-0003, Drying and Grinding of Soil and solid Samples

17.5.3 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil & Grease Samples for Radiochemical Analysis

17.5.4 ST-RC-5006, Decontamination of Laboratory Glassware. Labware and Equipment
17.5.5 ST-RD-0403, Low Background Gas Flow Proportional Counting (GFPC) System
17.5.6 ST-QA-0002, Standards and Reagent Preparation
17.5.7 ST-QA-0005, Calibration and Verification Procedure for Laboratory Support Equipment
17.5.8 ST-QA-0036, Non-conformance Memorandum (NCM) and Corrective Action Processes
17.5.9 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Modifictions to method C1507-07:
- 18.1.1 The reference method is geared for 10 g sample aliquot. This SOP is scaled for smaller aliquot volumes as needed.
- 18.1.2 Total dissolution with HF is only performed for samples expected to contain refractory strontium or when required by the client.
- 18.1.3 A 3 M nitric acid/0.05 M oxalic acid rinse is used in the initial strontium portion of the procedure based upon extraction resin manufacturer (Eichrom Technologies, Inc) recommendation to remove possible interferences.
- 18.1.4 Based upon recommendation by Eichrom, boric acid is utilized following the HF digestion step to help destroy residual HF, which can interfere with the ensuing process.

18.2 Modifications to method SRW01:
- 18.2.1 The final load solution of 15 mL nitric acid/aluminum nitrate was changed to just 10 mL of 8M nitric acid to be consistent with the soil load solution.

19.0 CHANGES TO PREVIOUS REVISION

19.1 New procedure (no previous revision).

19.2 Rev. 1:
- 19.2.1 In section 11.2.8 updated the amounts needed to perform an ICP metals analysis for chemical yields.

19.3 Rev 2: (07/31/2014)
- 19.3.1 Grammatical errors fixed throughout SOP
- 19.3.2 Removed references to Quantims, replaced with LIMS
- 19.3.3 Updated text in Section 14

19.4 Annual Review – No Changes (04/04/2016)

19.5 Revision 3: (02/27/2017):
- 19.5.1 Updated section 5.0 – replaced MSDS with SDS
- 19.5.2 Updated section 7.0
  - 19.5.2.1 DI water source
  - 19.5.2.2 Clarified how to make reagents
- 19.5.3 Updated section 9
  - 19.5.3.1 Updated abbreviation for sample duplicate to match LIMS
  - 19.5.3.2 Removed duplicate paragraph from section 9.6
- 19.5.4 Updated section 11.0 – replaced Rad Capture with LIMS

19.6 Revision 4: (12/12/2017 Technical Review S. Bernsen/QA Review: M. Ward):
- 19.6.1 Updated section 7.4- added ICP Rinse/Diluent as a reagent.
- 19.6.2 Updated section 11.1.1- fixed the SOP from ST-RC-003 to ST-RC-0003.
- 19.6.3 Updated section 11.1.4- added 50 mL digestion tube
- 19.6.4 Updated section 11.2.2- added approximately 1 drop every 3 seconds.
- 19.6.5 Updated section 11.2.3- added approximately 1 drop every 1-2 seconds
- 19.6.6 Grammatical Errors Fixed

19.7 Revision 5: (11/9/2018 Technical Review: S. Bernsen, T. Romanko)

19.7.1 Updated signature page
19.7.2 Updated Title, Section 1 and Section 2 to add water to the matrices
19.7.3 Updated section 9.0- added calcium nitrate to method blank and LCS
19.7.4 Updated section 11.0
19.7.5 Added procedure for spiking and tracing samples prior to muffling
19.7.6 Updated digestion procedure to accommodate spiking and tracing prior to muffling
19.7.7 Added section 11.2 for water samples.
19.7.8 Clarified step 11.3.5 in the Yttrium 90 preparation.
19.7.9 Updated section18.

19.8 Added Eurofins logo and updated copyright information (4/16/2019)

19.9 Revision 6 (01/31/2020) Technical Review C. Mazariegos/ QA Review K. Ely
19.9.1 Removed references to quarts crucibles and ICP in section 6.0
19.9.2 Removed instructions for non-standardized Sr carriers in section 7.0
19.9.3 Generalized creation of MB and LCS samples and simplified requirements for spike amount in section 11.0
19.9.4 Removed instructions for Sr yield determination by ICP and added information on gravimentric carrier recovery determination in section 11.3
19.9.5 Corrected step references on Strontium Flow Chart
19.9.6 Added Yttrium Flow Chart (attachment 2)
19.9.7 Corrected spelling and grammar throughout
19.9.8 Section 9 – removed language pertaining to old LIMS system
19.9.9 Updated SOP names throughout.

19.10 Revision 7 (3/31/2021) Tech Review C. Mazariegos; QA Review M Ward/K. Ely
19.10.1 Added additional instructions (now standard prep) for alternative digestion of soils less than 2.5 g or solid samples that dissolve easily such as resin.
19.10.2 Corrected some spelling errors.
19.10.3 Adjusted step numbers to correlate to cahnges.

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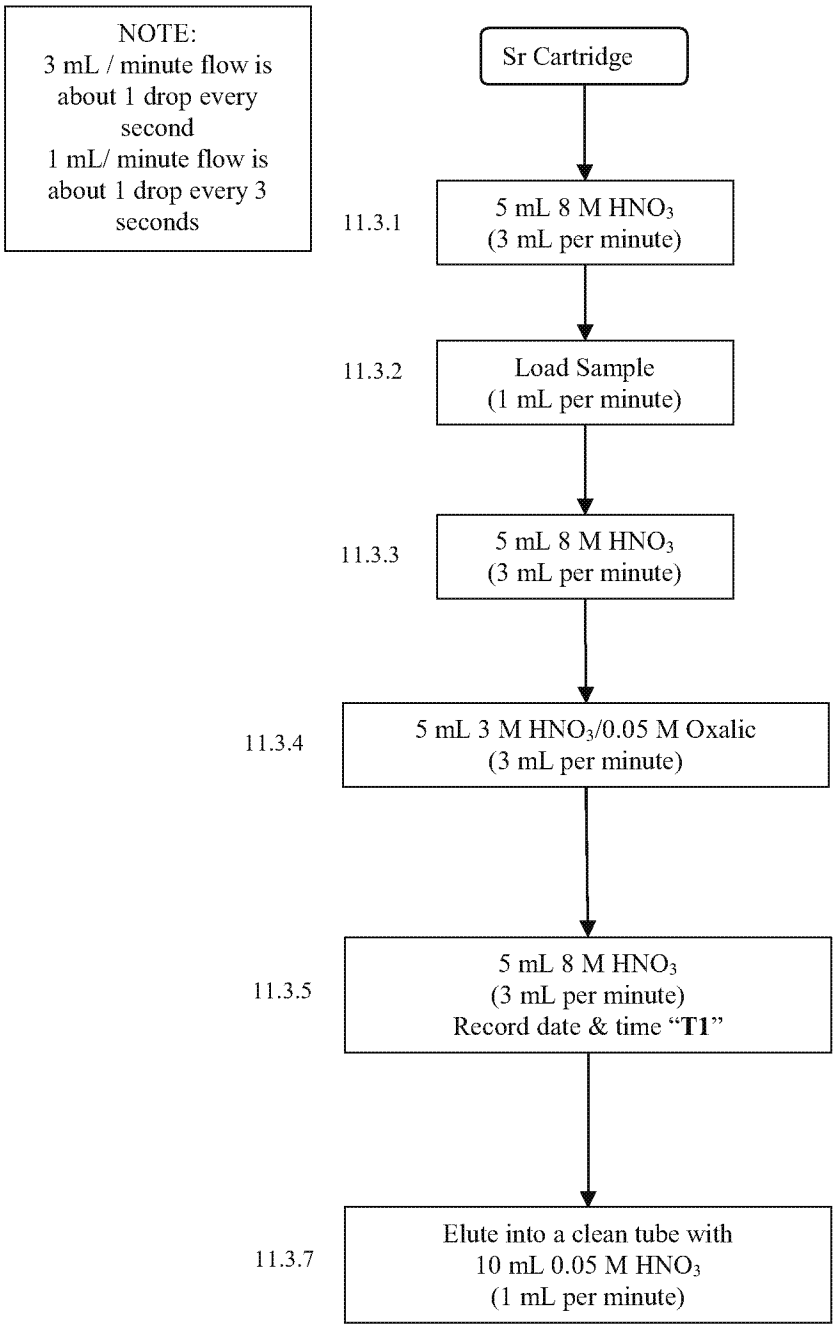
Attachment 1

Strontium via Sr-Specific Resin



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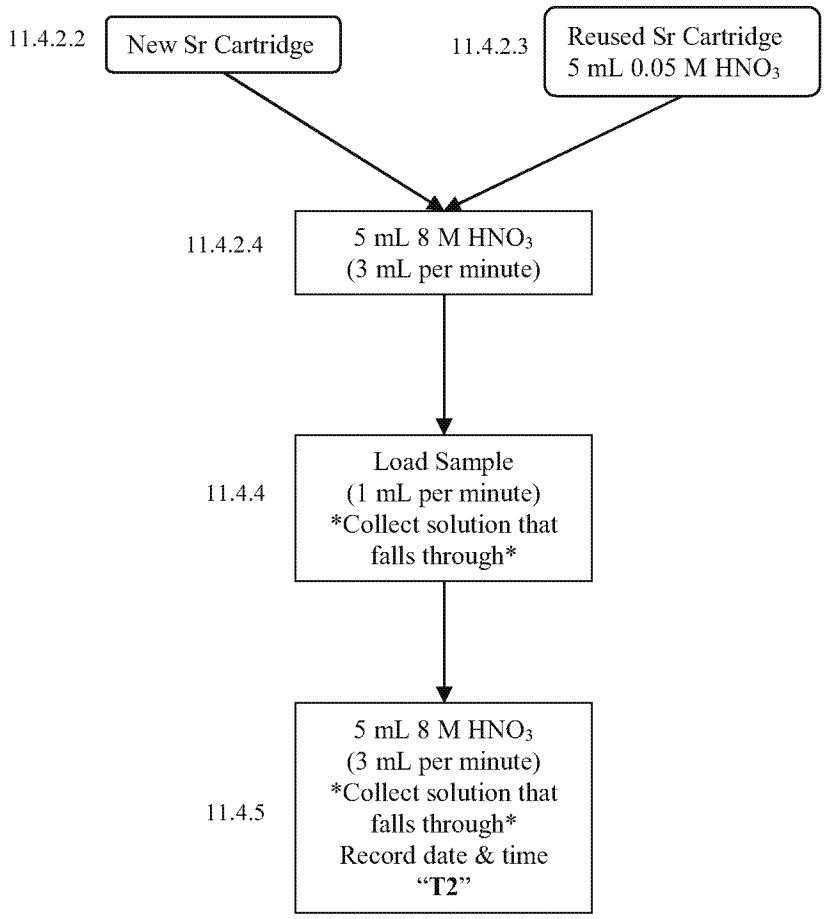
Attachment 2

Yttrium via Sr-Specific Resin



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CERTIFICATE OF ACCREDITATION

The ANSI National Accreditation Board

Hereby attests that

Eurofins TestAmerica, St. Louis Facility

13715 Rider Trail North
Earth City, Missouri 63045

Fulfills the requirements of

ISO/IEC 17025:2017

and the

U.S. Department of Defense (DoD) Quality Systems Manual for Environmental Laboratories (DoD QSM V5.3)

In the field of

TESTING

This certificate is valid only when accompanied by a current scope of accreditation document.
The current scope of accreditation can be verified at www.anab.org.

R. Douglas Leonard Jr., VP, PILR SBU

Expiry Date: 06 April 2022
Certificate Number: L2305

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

ANSI National Accreditation Board

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017 and U.S. DEPARTMENT OF DEFENSE (DoD) QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES (DoD QSM V5.3)

Eurofins TestAmerica, St. Louis Facility
13715 Rider Trail North
Earth City, Missouri 63045
Kristen Ely
314-298-8566

TESTING

Valid to: **April 6, 2022** Certificate Number: **L2305**

Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Aluminum
ICP-AES	EPA 6010B/6010C/6010D	Antimony
ICP-AES	EPA 6010B/6010C/6010D	Arsenic
ICP-AES	EPA 6010B/6010C/6010D	Barium
ICP-AES	EPA 6010B/6010C/6010D	Beryllium
ICP-AES	EPA 6010B/6010C/6010D	Bismuth
ICP-AES	EPA 6010B/6010C/6010D	Boron
ICP-AES	EPA 6010B/6010C/6010D	Cadmium
ICP-AES	EPA 6010B/6010C/6010D	Calcium
ICP-AES	EPA 6010B/6010C/6010D	Chromium
ICP-AES	EPA 6010B/6010C/6010D	Cobalt
ICP-AES	EPA 6010B/6010C/6010D	Copper
ICP-AES	EPA 6010B/6010C/6010D	Iron
ICP-AES	EPA 6010B/6010C/6010D	Lead
ICP-AES	EPA 6010B/6010C/6010D	Lithium
ICP-AES	EPA 6010B/6010C/6010D	Magnesium
ICP-AES	EPA 6010B/6010C/6010D	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Molybdenum
ICP-AES	EPA 6010B/6010C/6010D	Nickel
ICP-AES	EPA 6010B/6010C/6010D	Phosphorus
ICP-AES	EPA 6010B/6010C/6010D	Potassium
ICP-AES	EPA 6010B/6010C/6010D	Selenium
ICP-AES	EPA 6010B/6010C/6010D	Silicon
ICP-AES	EPA 6010B/6010C/6010D	Silver
ICP-AES	EPA 6010B/6010C/6010D	Sodium
ICP-AES	EPA 6010B/6010C/6010D	Strontium
ICP-AES	EPA 6010B/6010C/6010D	Sulfur
ICP-AES	EPA 6010B/6010C/6010D	Thallium
ICP-AES	EPA 6010B/6010C/6010D	Thorium
ICP-AES	EPA 6010B/6010C/6010D	Tin
ICP-AES	EPA 6010B/6010C/6010D	Titanium
ICP-AES	EPA 6010B/6010C/6010D	Vanadium
ICP-AES	EPA 6010B/6010C/6010D	Zinc
GC/MS	EPA 8260B/8260C/8260D	Acetone
GC/MS	EPA 8260B/8260C/8260D	Acetonitrile
GC/MS	EPA 8260B/8260C/8260D	Acrolein
GC/MS	EPA 8260B/8260C/8260D	Acrylonitrile
GC/MS	EPA 8260B/8260C/8260D	Benzene
GC/MS	EPA 8260B/8260C/8260D	Benzyl chloride
GC/MS	EPA 8260B/8260C/8260D	Bromobenzene
GC/MS	EPA 8260B/8260C/8260D	Bromochloromethane
GC/MS	EPA 8260B/8260C/8260D	Bromodichloromethane
GC/MS	EPA 8260B/8260C/8260D	Bromoform
GC/MS	EPA 8260B/8260C/8260D	Bromomethane
GC/MS	EPA 8260B/8260C/8260D	n-Butanol
GC/MS	EPA 8260B/8260C/8260D	2-Butanone
GC/MS	EPA 8260B/8260C/8260D	n-Butylbenzene
GC/MS	EPA 8260B/8260C/8260D	sec-Butylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	tert-Butylbenzene
GC/MS	EPA 8260B/8260C/8260D	Carbon disulfide
GC/MS	EPA 8260B/8260C/8260D	Carbon tetrachloride
GC/MS	EPA 8260B/8260C/8260D	Chlorobenzene
GC/MS	EPA 8260B/8260C/8260D	2-Chloro-1,3-butadiene
GC/MS	EPA 8260B/8260C/8260D	Chlorodibromomethane
GC/MS	EPA 8260B/8260C/8260D	Chloroethane
GC/MS	EPA 8260B/8260C/8260D	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/8260C/8260D	Chloroform
GC/MS	EPA 8260B/8260C/8260D	Chloromethane
GC/MS	EPA 8260B/8260C/8260D	Allyl chloride
GC/MS	EPA 8260B/8260C/8260D	2-Chlorotoluene
GC/MS	EPA 8260B/8260C/8260D	4-Chlorotoluene
GC/MS	EPA 8260B/8260C/8260D	Cyclohexane
GC/MS	EPA 8260B/8260C/8260D	Cyclohexanone
GC/MS	EPA 8260B/8260C/8260D	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dibromoethane
GC/MS	EPA 8260B/8260C/8260D	Dibromomethane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,3-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,4-Dichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	trans-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/8260C/8260D	Dichlorodifluoromethane
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloroethane
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloroethane
GC/MS	EPA 8260B/8260C/8260D	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloroethene
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloroethene (total)
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloropropane
GC/MS	EPA 8260B/8260C/8260D	1,3-Dichloropropane
GC/MS	EPA 8260B/8260C/8260D	2,2-Dichloropropane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	trans-1,3-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	1,1-Dichloropropene
GC/MS	EPA 8260B/8260C/8260D	1,2-Dichloro-1,1,2,2-tetrafluoroethane
GC/MS	EPA 8260B/8260C/8260D	Dimethyl disulfide
GC/MS	EPA 8260B/8260C/8260D	1,4-Dioxane
GC/MS	EPA 8260B/8260C/8260D	Ethyl acetate
GC/MS	EPA 8260B/8260C/8260D	Ethylbenzene
GC/MS	EPA 8260B/8260C/8260D	Ethyl ether
GC/MS	EPA 8260B/8260C/8260D	Ethyl methacrylate
GC/MS	EPA 8260B/8260C/8260D	Hexachlorobutadiene
GC/MS	EPA 8260B/8260C/8260D	n-Hexane
GC/MS	EPA 8260B/8260C/8260D	2-Hexanone
GC/MS	EPA 8260B/8260C/8260D	Iodomethane
GC/MS	EPA 8260B/8260C/8260D	Isobutanol
GC/MS	EPA 8260B/8260C/8260D	Isopropylbenzene
GC/MS	EPA 8260B/8260C/8260D	p-Isopropyltoluene
GC/MS	EPA 8260B/8260C/8260D	Methacrylonitrile
GC/MS	EPA 8260B/8260C/8260D	Methyl acetate
GC/MS	EPA 8260B/8260C/8260D	Methylcyclohexane
GC/MS	EPA 8260B/8260C/8260D	Methylene chloride
GC/MS	EPA 8260B/8260C/8260D	Methyl methacrylate
GC/MS	EPA 8260B/8260C/8260D	4-Methyl-2-pentanone
GC/MS	EPA 8260B/8260C/8260D	MTBE
GC/MS	EPA 8260B/8260C/8260D	Naphthalene
GC/MS	EPA 8260B/8260C/8260D	2-Nitropropane
GC/MS	EPA 8260B/8260C/8260D	Nonanal
GC/MS	EPA 8260B/8260C/8260D	Pentachloroethane
GC/MS	EPA 8260B/8260C/8260D	Propionitrile
GC/MS	EPA 8260B/8260C/8260D	n-Propylbenzene
GC/MS	EPA 8260B/8260C/8260D	Styrene
GC/MS	EPA 8260B/8260C/8260D	1,1,1,2-Tetrachloroethane

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Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/8260C/8260D	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/8260C/8260D	Tetrachloroethene
GC/MS	EPA 8260B/8260C/8260D	Tetrahydrofuran
GC/MS	EPA 8260B/8260C/8260D	Toluene
GC/MS	EPA 8260B/8260C/8260D	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/8260C/8260D	1,1,1-Trichloroethane
GC/MS	EPA 8260B/8260C/8260D	1,1,2-Trichloroethane
GC/MS	EPA 8260B/8260C/8260D	Trichloroethene
GC/MS	EPA 8260B/8260C/8260D	Trichlorofluoromethane
GC/MS	EPA 8260B/8260C/8260D	1,2,3-Trichloropropane
GC/MS	EPA 8260B/8260C/8260D	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/8260C/8260D	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/8260C/8260D	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B/8260C/8260D	Vinyl acetate
GC/MS	EPA 8260B/8260C/8260D	Vinyl chloride
GC/MS	EPA 8260B/8260C/8260D	m-Xylene & p-Xylene
GC/MS	EPA 8260B/8260C/8260D	o-Xylene
GC/MS	EPA 8260B/8260C/8260D	Xylenes (total)
GC/MS	EPA 8260B/8260C/8260D SIM	1,4-Dioxane
ICP-MS	EPA 6020/6020A/6020B	Aluminum
ICP-MS	EPA 6020/6020A/6020B	Antimony
ICP-MS	EPA 6020/6020A/6020B	Arsenic
ICP-MS	EPA 6020/6020A/6020B	Barium
ICP-MS	EPA 6020/6020A/6020B	Beryllium
ICP-MS	EPA 6020/6020A/6020B	Bismuth
ICP-MS	EPA 6020/6020A/6020B	Boron
ICP-MS	EPA 6020/6020A/6020B	Cadmium
ICP-MS	EPA 6020/6020A/6020B	Calcium
ICP-MS	EPA 6020/6020A/6020B	Cerium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Cesium
ICP-MS	EPA 6020/6020A/6020B	Chromium
ICP-MS	EPA 6020/6020A/6020B	Cobalt
ICP-MS	EPA 6020/6020A/6020B	Copper
ICP-MS	EPA 6020/6020A/6020B	Gold
ICP-MS	EPA 6020/6020A/6020B	Hafnium
ICP-MS	EPA 6020/6020A/6020B	Iron
ICP-MS	EPA 6020/6020A/6020B	Lanthanum
ICP-MS	EPA 6020/6020A/6020B	Lead
ICP-MS	EPA 6020/6020A/6020B	Lithium
ICP-MS	EPA 6020/6020A/6020B	Magnesium
ICP-MS	EPA 6020/6020A/6020B	Manganese
ICP-MS	EPA 6020/6020A/6020B	Molybdenum
ICP-MS	EPA 6020/6020A/6020B	Neodymium
ICP-MS	EPA 6020/6020A/6020B	Nickel
ICP-MS	EPA 6020/6020A/6020B	Niobium
ICP-MS	EPA 6020/6020A/6020B	Palladium
ICP-MS	EPA 6020/6020A/6020B	Phosphorus
ICP-MS	EPA 6020/6020A/6020B	Platinum
ICP-MS	EPA 6020/6020A/6020B	Potassium
ICP-MS	EPA 6020/6020A/6020B	Praseodymium
ICP-MS	EPA 6020/6020A/6020B	Rhenium
ICP-MS	EPA 6020/6020A/6020B	Rhodium
ICP-MS	EPA 6020/6020A/6020B	Ruthenium
ICP-MS	EPA 6020/6020A/6020B	Samarium
ICP-MS	EPA 6020/6020A/6020B	Selenium
ICP-MS	EPA 6020/6020A/6020B	Silver
ICP-MS	EPA 6020/6020A/6020B	Sodium
ICP-MS	EPA 6020/6020A/6020B	Strontium
ICP-MS	EPA 6020/6020A/6020B	Tantalum
ICP-MS	EPA 6020/6020A/6020B	Tellurium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Thallium
ICP-MS	EPA 6020/6020A/6020B	Thorium
ICP-MS	EPA 6020/6020A/6020B	Tin
ICP-MS	EPA 6020/6020A/6020B	Titanium
ICP-MS	EPA 6020/6020A/6020B	Tungsten
ICP-MS	EPA 6020/6020A/6020B	Uranium
ICP-MS	EPA 6020/6020A/6020B	Uranium 233
ICP-MS	EPA 6020/6020A/6020B	Uranium 234
ICP-MS	EPA 6020/6020A/6020B	Uranium 235
ICP-MS	EPA 6020/6020A/6020B	Uranium 236
ICP-MS	EPA 6020/6020A/6020B	Uranium 238
ICP-MS	EPA 6020/6020A/6020B	Vanadium
ICP-MS	EPA 6020/6020A/6020B	Yttrium
ICP-MS	EPA 6020/6020A/6020B	Zinc
ICP-MS	EPA 6020/6020A/6020B	Zirconium
ICP-MS	EPA 6020/6020A/6020B	Total Hardness
ICP-MS	EPA 6020/6020A/6020B	Dysprosium
ICP-MS	EPA 6020/6020A/6020B	Erbium
ICP-MS	EPA 6020/6020A/6020B	Europium
ICP-MS	EPA 6020/6020A/6020B	Gadolinium
ICP-MS	EPA 6020/6020A/6020B	Gallium
ICP-MS	EPA 6020/6020A/6020B	Holmium
ICP-MS	EPA 6020/6020A/6020B	Lutetium
ICP-MS	EPA 6020/6020A/6020B	Rubidium
ICP-MS	EPA 6020/6020A/6020B	Terbium
ICP-MS	EPA 6020/6020A/6020B	Thulium
ICP-MS	EPA 6020/6020A/6020B	Ytterbium
ICP-MS	EPA 200.8	Aluminum
ICP-MS	EPA 200.8	Antimony
ICP-MS	EPA 200.8	Arsenic
ICP-MS	EPA 200.8	Barium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Beryllium
ICP-MS	EPA 200.8	Boron
ICP-MS	EPA 200.8	Cadmium
ICP-MS	EPA 200.8	Calcium
ICP-MS	EPA 200.8	Cerium
ICP-MS	EPA 200.8	Cesium
ICP-MS	EPA 200.8	Chromium
ICP-MS	EPA 200.8	Cobalt
ICP-MS	EPA 200.8	Copper
ICP-MS	EPA 200.8	Gold
ICP-MS	EPA 200.8	Iron
ICP-MS	EPA 200.8	Lead
ICP-MS	EPA 200.8	Lithium
ICP-MS	EPA 200.8	Magnesium
ICP-MS	EPA 200.8	Manganese
ICP-MS	EPA 200.8	Molybdenum
ICP-MS	EPA 200.8	Nickel
ICP-MS	EPA 200.8	Phosphorus
ICP-MS	EPA 200.8	Platinum
ICP-MS	EPA 200.8	Potassium
ICP-MS	EPA 200.8	Rhodium
ICP-MS	EPA 200.8	Selenium
ICP-MS	EPA 200.8	Silver
ICP-MS	EPA 200.8	Sodium
ICP-MS	EPA 200.8	Strontium
ICP-MS	EPA 200.8	Tantalum
ICP-MS	EPA 200.8	Thallium
ICP-MS	EPA 200.8	Thorium
ICP-MS	EPA 200.8	Tin
ICP-MS	EPA 200.8	Titanium
ICP-MS	EPA 200.8	Tungsten

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Uranium
ICP-MS	EPA 200.8	Vanadium
ICP-MS	EPA 200.8	Zinc
ICP-MS	EPA 200.8	Zirconium
ICP-AES	EPA 200.7	Aluminum
ICP-AES	EPA 200.7	Antimony
ICP-AES	EPA 200.7	Arsenic
ICP-AES	EPA 200.7	Barium
ICP-AES	EPA 200.7	Beryllium
ICP-AES	EPA 200.7	Bismuth
ICP-AES	EPA 200.7	Boron
ICP-AES	EPA 200.7	Cadmium
ICP-AES	EPA 200.7	Calcium
ICP-AES	EPA 200.7	Chromium
ICP-AES	EPA 200.7	Cobalt
ICP-AES	EPA 200.7	Copper
ICP-AES	EPA 200.7	Iron
ICP-AES	EPA 200.7	Lead
ICP-AES	EPA 200.7	Lithium
ICP-AES	EPA 200.7	Magnesium
ICP-AES	EPA 200.7	Manganese
ICP-AES	EPA 200.7	Molybdenum
ICP-AES	EPA 200.7	Nickel
ICP-AES	EPA 200.7	Phosphorus
ICP-AES	EPA 200.7	Potassium
ICP-AES	EPA 200.7	Selenium
ICP-AES	EPA 200.7	Silicon
ICP-AES	EPA 200.7	Silver
ICP-AES	EPA 200.7	Sodium
ICP-AES	EPA 200.7	Strontium
ICP-AES	EPA 200.7	Sulfur

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Non-Potable Water		
Technology	**Method**	**Analyte**
ICP-AES	EPA 200.7	Thallium
ICP-AES	EPA 200.7	Thorium
ICP-AES	EPA 200.7	Tin
ICP-AES	EPA 200.7	Titanium
ICP-AES	EPA 200.7	Vanadium
ICP-AES	EPA 200.7	Zinc
CVAA	EPA 245.1/7470A	Mercury
Ion Chromatrography	EPA 300.0/9056/9056A	Bromide
Ion Chromatrography	EPA 300.0/9056/9056A	Chloride
Ion Chromatrography	EPA 300.0/9056/9056A	Fluoride
Ion Chromatrography	EPA 300.0/9056/9056A	Nitrate
Ion Chromatrography	EPA 300.0/9056/9056A	Nitrite
Ion Chromatrography	EPA 300.0/9056/9056A	Sulfate
Ion Chromatrography	EPA 300.0/9056/9056A	Ortho-phosphate
Ion Chromatrography	EPA 300.0/9056/9056A	Iodide
Probe	EPA 9040C EPA 150.1 SM 4500-H+ B -2011	pH
Colormetric	EPA 7196A	Hex Chromium
Gas Flow Proportional Counter	EPA 900.0 EPA 9310 SM 7110C	gross alpha/beta
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02 DOE HASL 300 Sr-03	Strontium-90
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222

Non-Potable Water		
Technology	**Method**	**Analyte**
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01 HASL 300 Tc-02-RC	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141

Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212

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Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46

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Non-Potable Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R DOE HASL 300 U-02-RC	Isotopic Uranium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium

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Non-Potable Water		
Technology	**Method**	**Analyte**
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Alpha Spectroscopy	ST-RC-0301	Radium-226
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Extraction Chromatography	ST-RC-0058	Strontium-90
Preparation	**Method**	**Type**
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Acid Digestion (Aqueous samples)	EPA 3010A EPA 3005A	Acid Digestion for Metals (Aqueous samples)
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction

Drinking Water		
Technology	**Method**	**Analyte**
ICP-MS	EPA 200.8	Uranium
Alpha Spectroscopy	DOE HASL 300 U-02-RC	Isotopic Uranium
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	Gross alpha/beta
Gas Flow Proportional Counter	SM 7110C	Gross alpha

Drinking Water		
Technology	**Method**	**Analyte**
Gas Flow Proportional Counter	ST-RC-0036 ST-RD-0403	Chlorine-36
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02	Strontium-90
Liquid Scintillation Counter	SM 7500-Rn B	Radon-222
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147

Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231

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Drinking Water		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Aluminum
ICP-AES	EPA 6010B/6010C/6010D	Antimony
ICP-AES	EPA 6010B/6010C/6010D	Arsenic
ICP-AES	EPA 6010B/6010C/6010D	Barium
ICP-AES	EPA 6010B/6010C/6010D	Beryllium
ICP-AES	EPA 6010B/6010C/6010D	Bismuth
ICP-AES	EPA 6010B/6010C/6010D	Boron
ICP-AES	EPA 6010B/6010C/6010D	Cadmium
ICP-AES	EPA 6010B/6010C/6010D	Calcium
ICP-AES	EPA 6010B/6010C/6010D	Chromium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-AES	EPA 6010B/6010C/6010D	Cobalt
ICP-AES	EPA 6010B/6010C/6010D	Copper
ICP-AES	EPA 6010B/6010C/6010D	Iron
ICP-AES	EPA 6010B/6010C/6010D	Lead
ICP-AES	EPA 6010B/6010C/6010D	Lithium
ICP-AES	EPA 6010B/6010C/6010D	Magnesium
ICP-AES	EPA 6010B/6010C/6010D	Manganese
ICP-AES	EPA 6010B/6010C/6010D	Molybdenum
ICP-AES	EPA 6010B/6010C/6010D	Nickel
ICP-AES	EPA 6010B/6010C/6010D	Phosphorus
ICP-AES	EPA 6010B/6010C/6010D	Potassium
ICP-AES	EPA 6010B/6010C/6010D	Selenium
ICP-AES	EPA 6010B/6010C/6010D	Silicon
ICP-AES	EPA 6010B/6010C/6010D	Silver
ICP-AES	EPA 6010B/6010C/6010D	Sodium
ICP-AES	EPA 6010B/6010C/6010D	Strontium
ICP-AES	EPA 6010B/6010C/6010D	Sulfur
ICP-AES	EPA 6010B/6010C/6010D	Thallium
ICP-AES	EPA 6010B/6010C/6010D	Thorium
ICP-AES	EPA 6010B/6010C/6010D	Tin
ICP-AES	EPA 6010B/6010C/6010D	Titanium
ICP-AES	EPA 6010B/6010C/6010D	Vanadium
ICP-AES	EPA 6010B/6010C/6010D	Zinc
ICP-MS	EPA 6020/6020A/6020B	Aluminum
ICP-MS	EPA 6020/6020A/6020B	Antimony
ICP-MS	EPA 6020/6020A/6020B	Arsenic
ICP-MS	EPA 6020/6020A/6020B	Barium
ICP-MS	EPA 6020/6020A/6020B	Beryllium
ICP-MS	EPA 6020/6020A/6020B	Bismuth
ICP-MS	EPA 6020/6020A/6020B	Boron
ICP-MS	EPA 6020/6020A/6020B	Cadmium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Calcium
ICP-MS	EPA 6020/6020A/6020B	Cerium
ICP-MS	EPA 6020/6020A/6020B	Cesium
ICP-MS	EPA 6020/6020A/6020B	Chromium
ICP-MS	EPA 6020/6020A/6020B	Cobalt
ICP-MS	EPA 6020/6020A/6020B	Copper
ICP-MS	EPA 6020/6020A/6020B	Gold
ICP-MS	EPA 6020/6020A/6020B	Hafnium
ICP-MS	EPA 6020/6020A/6020B	Iron
ICP-MS	EPA 6020/6020A/6020B	Lanthanum
ICP-MS	EPA 6020/6020A/6020B	Lead
ICP-MS	EPA 6020/6020A/6020B	Lithium
ICP-MS	EPA 6020/6020A/6020B	Magnesium
ICP-MS	EPA 6020/6020A/6020B	Manganese
ICP-MS	EPA 6020/6020A/6020B	Molybdenum
ICP-MS	EPA 6020/6020A/6020B	Neodymium
ICP-MS	EPA 6020/6020A/6020B	Nickel
ICP-MS	EPA 6020/6020A/6020B	Niobium
ICP-MS	EPA 6020/6020A/6020B	Palladium
ICP-MS	EPA 6020/6020A/6020B	Phosphorus
ICP-MS	EPA 6020/6020A/6020B	Platinum
ICP-MS	EPA 6020/6020A/6020B	Potassium
ICP-MS	EPA 6020/6020A/6020B	Praseodymium
ICP-MS	EPA 6020/6020A/6020B	Rhenium
ICP-MS	EPA 6020/6020A/6020B	Rhodium
ICP-MS	EPA 6020/6020A/6020B	Ruthenium
ICP-MS	EPA 6020/6020A/6020B	Samarium
ICP-MS	EPA 6020/6020A/6020B	Selenium
ICP-MS	EPA 6020/6020A/6020B	Silver
ICP-MS	EPA 6020/6020A/6020B	Sodium
ICP-MS	EPA 6020/6020A/6020B	Strontium

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP-MS	EPA 6020/6020A/6020B	Tantalum
ICP-MS	EPA 6020/6020A/6020B	Tellurium
ICP-MS	EPA 6020/6020A/6020B	Thallium
ICP-MS	EPA 6020/6020A/6020B	Thorium
ICP-MS	EPA 6020/6020A/6020B	Tin
ICP-MS	EPA 6020/6020A/6020B	Titanium
ICP-MS	EPA 6020/6020A/6020B	Tungsten
ICP-MS	EPA 6020/6020A/6020B	Uranium
ICP-MS	EPA 6020/6020A/6020B	Uranium 233
ICP-MS	EPA 6020/6020A/6020B	Uranium 234
ICP-MS	EPA 6020/6020A/6020B	Uranium 235
ICP-MS	EPA 6020/6020A/6020B	Uranium 236
ICP-MS	EPA 6020/6020A/6020B	Uranium 238
ICP-MS	EPA 6020/6020A/6020B	Vanadium
ICP-MS	EPA 6020/6020A/6020B	Yttrium
ICP-MS	EPA 6020/6020A/6020B	Zinc
ICP-MS	EPA 6020/6020A/6020B	Zirconium
CVAA	EPA 7471A/7471B	Mercury
Ion Chromatrography	EPA 300.0/9056A	Bromide
Ion Chromatrography	EPA 300.0/9056A	Chloride
Ion Chromatrography	EPA 300.0/9056A	Fluoride
Ion Chromatrography	EPA 300.0/9056A	Nitrate
Ion Chromatrography	EPA 300.0/9056A	Nitrite
Ion Chromatrography	EPA 300.0/9056A	Sulfate
Ion Chromatrography	EPA 300.0/9056A	Ortho-phosph
Ion Chromatrography	EPA 300.0/9056A	Iodide
Probe	EPA 9045D	pH
Gas Flow Proportional Counter	EPA 900.0 EPA 9310	gross alpha/beta
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	Radium-226
Gas Flow Proportional Counter	EPA 903.0 EPA 9315	total radium

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gas Flow Proportional Counter	EPA 904.0 EPA 9320	Radium-228
Gas Flow Proportional Counter	EPA 905.0 DOE HASL 300 Sr-02 DOE HASL 300 Sr-03	Strontium-90
Liquid Scintillation Counter	ST-RC-0079	Selenium-79
Liquid Scintillation Counter	EPA 906.0	Tritium
Liquid Scintillation Counter	Eichrom Technologies TCW01/TCS01 HASL 300 Tc-02-RC	Tecnetium-99
Liquid Scintillation Counter	EERF C-01-C14	Carbon-14
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Gamma Emitters:
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 227 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Actinium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Americium 241
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 124
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Antimony 125
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium/Lanthanum-140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 133
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Barium 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Beryllium 7
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 211 eq Th-227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 207
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth-210M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 212

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Bismuth 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 141
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 139
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cerium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 134
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cesium 137
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 56
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 57
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 58
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Cobalt 60
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 152
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 154
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Europium 155
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Hafnium 181
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iodine 131
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iridium 192
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Iron 59
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lanthanum 140
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 210
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 211

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 212
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Lead 214
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Manganese 54
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Mercury 203
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 237
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Neptunium 239
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 94
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Niobium 95
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Potassium 40
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 144
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 146
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Promethium 147
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234M
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Protactinium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium (226)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 223 (assumes equilibrium w/ Th-227)
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Radium 224
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Ruthenium 106

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Scandium 46
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 22
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Sodium 24
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Strontium 85
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thallium 208
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 227
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 228
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 230
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 231
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 232
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Thorium 234
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Tin 113
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 235
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Uranium 238
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Vanadium-48
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Yttrium 88
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zinc 65
Gamma Spectroscopy	EPA 901.1 / DOE HASL 300 Ga-01-R	Zirconium 95
Alpha Spectroscopy	DOE HASL 300 A-01-R	Alpha spec analysis:
Alpha Spectroscopy	DOE HASL 300 A-01-R DOE HASL 300 U-02-RC	Isotopic Uranium

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Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Thorium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Americium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Plutonium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Neptunium
Alpha Spectroscopy	DOE HASL 300 A-01-R	Isotopic Curium
Alpha Spectroscopy	ST-RC-0301	Radium-226
Liquid Scintillation Counter	Eichrom Technologies OTW01, OTS01	Lead-210
Alpha Spectroscopy	Laboratory SOP ST-RC-0210	Polonium-210
Liquid Scintillation Counter	Eichrom Technologies FEW01	Iron-55
Liquid Scintillation Counter	DOE RP-300	Nickel 59/63
Liquid Scintillation Counter	SM 7500-IB	Iodine-129
Preparation	**Method**	**Type**
Volatile Prep	EPA 5000	Sample Preparation for Volatile Organic Compounds
Acid Digestion (Aqueous samples)	EPA 3010A	Acid Digestion for Metals (Aqueous samples)
Acid Digestion (solids)	EPA 3050B	Acid Digestion for Metals of Sedimtent/Soils
Purge & Trap	EPA 5030C	Purge & Trap for Aqueous Volatile Samples
Closed System Purge & Trap and Extraction for Volatiles	EPA 5035A	Closed System Purge & Trap and Extraction for Volatiles
TCLP Extraction	EPA 1311	TCLP Extraction
SPLP Extraction	EPA 1312	SPLP Extraction
CWET Extraction	CA Title 22	CWET Extraction
Extraction Chromatography	Eichrom Technologies FEW01	Iron-55
Extraction Chromatography	ST-RC-0058	Strontium-90

Note:

1. This scope is formatted as part of a single document including Certificate of Accreditation No. L2305.

R. Douglas Leonard Jr., VP, PILR SBU

Title: ISOTOPIC THORIUM, PLUTONIUM AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESINS

Approvals (Signature/Date):

09/05/2019 Chelsea Mazariegos — Date Radiochemistry Prep Manager	9/9/19 Michael Rideshower — Date Health & Safety Manager / Coordinator
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1.0 SCOPE AND APPLICATION

1.1 This SOP provides a rapid, reliable method for separation of Thorium, Plutonium and Uranium in various matrices.
- 1.1.1 If only Uranium analysis is requested, see SOP: ST-RC-0238.
- 1.1.2 If other actinides in addition to Thorium, Plutonium and Uranium are requested, please see SOP index to determine applicable SOP.

1.2 This SOP is based on:
- 1.2.1 Eichrom Technologies, Inc. Analytical Procedures "ACW13 VBS Thorium, Plutonium and Uranium in Water (with Vacuum Box System)" and "ACW01 Uranium and Thorium in Water".
- 1.2.2 HASL-300 EML Procedure Manual

1.3 This procedure is applicable to water, soil, filter, biota, and oil.
- 1.3.1 Soil, filter, biota and oils are pre-prepared in accordance with SOP, ST-RC-0004.
- 1.3.2 Water preparation is contained within this SOP.

1.4 The requested limits, minimum detection amounts and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for separation of Thorium, Plutonium and Uranium using Eichrom resin prior to measurement by alpha spectrometry. A calcium phosphate precipitation technique is used to concentrate and remove actinides from water samples. Soils, Sludge and Filters are prepared for analysis using ST-RC-0004. Tracers are used to correct for chemical recovery and correct results to improve precision and accuracy.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.

3.2 Tracer - A known amount of ^{232}Uranium, ^{229}Thorium, ^{242}Plutonium, (or ^{236}Plutonium), added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

4.0 INTERFERENCES

4.1 Actinides with unresolvable alpha energies, such as Americium-241 and Plutonium-238, must be chemically separated to enable measurement of the target actinide(s). This method separates these isotopes effectively.

4.2 Samples that are high in carbonates and phosphates, as indicated by a violent and vigorous reaction during the initial phases of digestion, need to be loaded in a minimum of 40 mL of load solution. The increased amount of load increases the amount of aluminum nitrate that the samples are exposed to. The extra aluminum nitrate helps to bind phosphates which interfere with thorium uptake.

4.3 Neptunium-237 can interfere with the Plutonium-242. This interference can be avoided by increasing the normality of the hydrochloric acid rinse and increasing the concentration of the titanium trichloride eluant.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.1.1 None.

5.2 PRIMARY MATERIALS USED

5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm (TWA)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Calcium nitrate	Oxidizer	None established	Inhalation symptoms include coughing and shortness of breath. Skin contact symptoms include redness, itching, and pain. Eye contact causes irritation, redness and pain.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
Lead nitrate	Poison Oxidizer	0.05 mg/m^3 (TWA)	Inhalation of lead can produce local irritation of bronchia and lungs with acute exposure causing a metallic taste in the mouth and chest and abdominal pain. Ingestion symptoms can include abdominal pain and spasms, nausea, vomiting, and headache. Absorption through skin can occur causing symptoms similar to ingestion. Skin contact may cause local irritation, redness and pain. Absorption can also occur through eye tissue.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.

Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m^3 (TWA)	Inhalation may cause irritation of the nose and throat, and labored breathing. Skin contact symptoms include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain, and severe tissue burns.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short term exposure limit			
Ceiling – At no time should this exposure limit be exceeded.			

6.0 EQUIPMENT AND SUPPLIES

6.1 Beakers, 50-2000 mL

6.2 Analytical balance - 0.0001 g sensitivity

6.3 Centrifuge

6.4 Centrifuge tubes, poly, 50 mL with cap

6.5 Pipettes, glass or plastic, disposable

6.6 Pipettes, mechanical

6.7 Fume hood

6.8 Hotplate

6.9 Vortex mixer

6.10 pH strips, narrow range

6.11 Vacuum Box, Eichrom part number AC-24-BOX, or equivalent

6.12 Syringe filter, 25 mm acrodisc, 0.45 or 0.70 µm

6.13 Cartridge reservoirs/syringe/funnel-20 mL B-D Luer Lok syringe Part Number 301625 (Fisher part number 14-823-2B), or equivalent.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water

7.3 Aluminum Nitrate, solid.

7.4 Ammonium hydrogen phosphate dibasic (3.2M)

7.4.1 Dissolve 104 g of $(NH_4)_2HPO_4$ in 200 mL of water, heat gently to dissolve and dilute to 250 mL with water.

7.5 Ammonium hydroxide (NH4OH), Reagent.

7.6 L (+) Ascorbic Acid, reagent powder.
7.6.1 L (+) Ascorbic Acid solution, 2.5 g dissolved in 10 mL of DI water.

7.7 Bromocresol Purple indicator solution
7.7.1 Dissolve 0.20 g of Bromocresol Purple (520.24 F.W.) in 250 mL of water, add 1 mL of concentrated Ammonium Hydroxide.

7.8 Calcium nitrate (1.25M)
7.8.1 Dissolve 51 g of $Ca(NO_3)_2$ in 100 mL of water and dilute to 250 mL with water.

7.9 Hydrochloric acid (12M) - concentrated HCl (sp gr 1.19).
7.9.1 Hydrochloric acid (9M) Non-Critical reagent - Add approximately 1500 mL of concentrated HCl (sp gr 1.19) to approximately 200 mL of water and dilute to 2 liters.
7.9.2 Hydrochloric acid (6M) Non-Critical reagent – In a pre-m,arked 2.5 L bottle, add approximately 1000 mL of concentrated HCl (sp gr 1.19) to approximately 200 mL of water and dilute to 2 L with DI water.
7.9.3 Hydrochloric acid (1M) Non-Critical reagent - Add approximately 167 mL of concentrated HCl (sp gr 1.19) to approximately 200 mL of water and dilute to 2 L with DI water.

7.10 Hydrochloric acid (5M) - 0.05M oxalic acid solution (Non-Critical reagent):
7.10.1 Add 12.6 grams of oxalic acid dihydrate in approximately 800 mL of water. Add approximately 834 mL of concentrated hydrochloric acid. Dilute to 2 L with DI water, add a stir bar, and place on stir plate until oxalic acid is completely dissolved

7.11 Lead Nitrate (Reagent, crystals).
7.11.1 Lead Nitrate 1 % wt/vol. solution. Dissolve 1 g of lead nitrate crystals in 100 mL of DI water.

7.12 Nitric acid (16M) - concentrated HNO_3 (sp gr 1.42).
7.12.1 Nitric acid (3M) Non-Critical reagent – Add Approximately 375 mL of concentrated nitric acid to approximately 1500 mL of DI water and dilute to 2 L.

7.13 Load solution [Nitric acid (3 M) - aluminum nitrate (1 M)] Non-Critical reagent:
7.13.1 Weigh 1500 g $Al(NO_3)_3 \cdot 9H_2O$ in a 4 liter beaker. Add approximately 800 mL of water to a 4 L beaker. Add approximately 764 mL of concentrated nitric acid. Dilute to 4 L with DI water. Add stir bar, cover with watch glass and place on astir plate until aluminum nitrate is completely dissolved.

7.14 Potassium Hydroxide, KOH

7.15 Potassium Sulfate (Reagent Crystals).

7.16 Oxalic Acid, reagent, crystals.

7.17 Sodium Nitrite, $NaNO_2$ reagent crystals.
7.17.1 Sodium Nitrite solution– dissolve 1.0 grams of sodium nitrite crystals in 10 mL of DI water.

7.18 Titanium trichloride, $TiCl_3$, 10% or 20% solution, commercially available.

7.19 TEVA Resin - prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.

7.20 UTEVA Resin-prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.

7.21 Plutonium-242 tracer standard, 10-20 dpm/mL (Plutonium-236 can also be used).

7.22 Plutonium-238 and/or Plutonium-239 spike standard.

7.23 Thorium-229 tracer standard , 10-20 dpm/mL.

7.24 Natural Thorium spike standard (Thorium-232/Thorium-228), approximately 10-20 dpm/mL.

7.25 Thorium-230 spike standard, approximately 10-20 dpm/mL.

7.26 TRM solid reference material.

7.27 Uranium-232 tracer, approximatly 10-20dpm/mL.

7.27.1 Clean uranium free of Th-228 daughter, removed by lead sulfate precipitation, activity verified prior to use. A Th-228 free Uranium-232 standard may be made as descirbed below.

7.27.2 Dilute the appropriate aliquot of stock to about 40mL with DI water.

7.27.3 Add 3 grams of potassium sulfate.

7.27.4 Adjust the pH to 1.5 using narrow range pH strips with either 2M H_2SO_4 or 2M KOH.

7.27.5 While mixing, slowly add 25 mL of 1% $Pb(NO_3)_2$.

7.27.6 Adjust the pH to 1.5 using narrow range pH strips with either 2M H_2SO_4 or 2M KOH.

7.27.7 Dilute to 100 mL with water, and mix well. Solution should be spun (at a rate fast enough to form a vortex) continuously for at least 30 minutes to remove any Thorium that maybe in solution.

7.27.8 Let stand for at least 1 hour. Centrifuge the solution for 30 minutes. Use the clean U-232 solution as soon as possible after removing the Th-228.

7.27.9 Before each use, shake the standard at least 30 minutes (to absorb any ingrown Th-228 onto the sulfate precipitate), and let the precipitate settle (centrifuge). Do not disturb the precipitate while using the standard.

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples are checked upon receipt by sample control, therefore, the pH does not require checking prior to analysis.

8.3.1.1 Aqueous samples acidified upon receipt (designated by label on the bottle) do require checking the pH prior to analysis.

8.4 Solid sample requirements are found in SOP ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Actinide Analysis".

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same reagent lots. Where no preparation method exists (e.g. water samples for volatile organics, water samples for anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, reagent lots and personnel.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate (DU), an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water with Nitric Acid.

9.2.4 For non-aqueous analyses,the method blank is comprised of 1.25M Calcium Nitrate. See the soil preparation SOP ST-RC-0004.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water with Nitric acid fortified with the isotopes of interest.

9.3.4 For non-aqueous analyses, the LCS is comprised of a TRM sold reference material or 1.25M Calcium Nitrate fortified with the isotopes of interest. See the soil prep SOP ST-RC-0004.

9.3.4.1 For Am, Cm, Pu, Uranium only, use Calcium nitrate

9.3.4.2 For Thorium use the TRM standard.

9.4 **Matrix Spike(MS)/Matrix Spike Duplicate(MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (DU)**

9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and pipette calibrations must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

11.0 PROCEDURE

11.1 **For NON-AQUEOUS matrices (soil, oil, biota, etc) see SOP: ST-RC-0004 for initial sample preparation and proceed to section 11.5 of this SOP.**

11.2 Water Sample Preparation:

- 11.2.1 Prepare method blank and LCS using 500 mL or 1000 mL DI water (match to volume of associated samples).
  - 11.2.1.1 Acidify with nitric acid to a pH < 2.
- 11.2.2 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.
- 11.2.3 Weigh approximately 500 mL or 1000 mL (depending on the dection limit) of sample into an appropriate size beaker. Record weight in LIMS.
  - 11.2.3.1 Aqueous sample aliquot volumes are determined by mass and use an assumed density of 1 g/mL.
  - 11.2.3.2 If upon visual inspection a sample is suspected to have a high density (>1.2g/mL, e.g. brine or waste) or a low density (<0.98g/mL, e.g. mixed solvent) the sample density will be measured and the volume determined arithemetically (sample mass divided by density equals volume).
- 11.2.4 Add appropriate tracers or standards. Generally 10 – 20 dpm of each of the Thorium, Uranium and/or Plutonium tracers are added. (Typically 0.1 mL or 0.2 mL depending on the reporting limit)
  - 11.2.4.1 Spike LCS and MS (if applicable) with isotopes of interest. (Typically 0.1 mL or 0.2 mL depending on the reporting limit)

11.3 Calcium phosphate precipitation:

- 11.3.1 Pipette 0.5 mL of 1.25M $Ca(NO_3)_2$ to each beaker.
- 11.3.2 Pipette 0.2 mL of 3.2 M $(NH_4)_2HPO_4$ solution to each beaker .
- 11.3.3 Add 3-5 drops Bromocresol Purple indicator to each beakerPlace beakers on a hot plate.
- 11.3.4 Allow the samples to heat to near boiling approximately 30 minutes.
- 11.3.5 Once the samples reach near boiling, turn the heat down to medium.
- 11.3.6 Add enough concentrated NH_4OH with a squirt bottle to reach the bromocresol purple indicator end point and form $Ca_3(PO_4)_2$ precipitate. Stir samples with a disposable transfer pipetteAllow the sample to heat for another 20-30 minutes.
- 11.3.7 Remove from the hot plate, allow sample to cool and precipitate to settle.
- 11.3.8 Decant to base waste.
- 11.3.9 Transfer the precipitate to a centrifuge tube and centrifuge the precipitate for approximately 5 minutes at 2000 rpm.
- 11.3.10 Decant supernatant and discard to base waste.
- 11.3.11 Proceed to section 11.5

11.4 Evaporation (Alternative option to Calcium Phosphate precipitation):

- 11.4.1 This option may be used when large sample volumes are needed to achieve low level reporting limits.
  - 11.4.1.1 Consult Manager/Supervisor to determine when this option should be used.
- 11.4.2 Evaporate sample on a hot plate to less than 50 mL and transfer to a 100 mL beaker.
  - 11.4.2.1 Note: For some water samples, calcium sulfate formation may occur during evaporation. If this occurs, use the calcium phosphate precipitation option in step 11.4
- 11.4.3 Gently evaporate the sample to dryness and redissolve in approximately 5 mL of concentrated HNO_3 (sp gr 1.42). Repeat step two more times, evaporate to dryness and proceed to step 11.5.

11.5 Thorium/Plutonium/Uranium Separation using Eichrom resins.

11.5.1 Dissolve calcium phosphate precipitate, soil dissolution residue or evaporated water sample with 15 mL of Load solution..

11.5.1.1 **For waters**:

11.5.1.1.1 A additional 5 mL load solution aliqouts may be necessary to dissolve the sample residue. Do not use more than 30 mL of load solution.

NOTE: Samples that are high in carbonates and phosphates, as indicated by a violent and /or vigorous reaction during the initial phases of digestion, need to be loaded in a minimum of 40 mL of load solution.

11.5.1.1.2 If particles are observed or the solution is cloudy, the samples can be centrifuged at approximately 2000 rpm for 5 minutes.

NOTE: The use of filtration is also permitted, e.g. syringe filter if the solution is still cloudy.

11.5.2 For each sample dissolved in load solution, place a UTEVA resin cartridge in the vacuum box. Lock a TEVA Resin cartridge onto the top of the UTEVA cartridge. Attach a plastic syringe funnel to the top of the TEVA cartidge.

11.5.2.1 For batches that are being performed sequentially with stacked cartridges, the LCS (for the analyte that is taken from the lower cartridge) should be treated the same as the samples.

11.5.2.1.1 Ex: the LCS for a uranium sample, being run sequentially with thorium, will be loaded through a TEVA/UTEVA stacked cartridge.

11.5.2.2 For single analyte analyses, cartridges that are below the cartridge used to bind the analyte of interest, are NOT required to be utilized.

11.5.2.2.1 Ex: For a plutonium/thorium LCS, the UTEVA cartridge would be omitted.

11.5.3 Place a waste collection reservoir inside the vacuum box to catch the column effluent.

11.5.4 Just prior to loading the sample condition the resin:

11.5.4.1 Turn on the vacuum pump.

11.5.4.2 Add 5 mL of 3M HNO_3 into each funnel.

11.5.4.3 Allow the solution to be pulled through the columns by adjusting the flow rate on top of the vacuum box.

11.5.4.4 The flow rate should be approximately 3 mL per minute. Discard effluent to waste.

NOTE: Approximately 20 drops equals 1 mL. Adjust the flow for each solution added.

11.5.5 For samples requiring Plutonium analysis (If samples do not require Plutonium, proceed to step 11.5.8):

11.5.5.1 Add 1 mL of ascorbic acid to the sample load solution in the centrifuge tube . Heat in hot water bath for approximately 5 minutes.

11.5.5.2 Add 1 mL of $NaNO_2$ solution to the sample load solution in the centrifuge tube and heat in hot water bath for approximately 5 minutes.

11.5.5.3 Remove samples from hot water bath and let cool in cold water bath until samples are at or slightly below room temperature.

11.5.6 Syringe Filter each sample load solution into the appropriate TEVA/UTEVA Resin cartridge funnel. Allow to drain. Adjust the flow rate to approximately 1 mL per minute.

NOTE: the TEVA and the UTEVA cartridge can turn blueish green as the load solution drains through it.

11.5.7 Rinse the funnel with 20 mL of 3M HNO_3 and allow to drain. Adjust flow to approximately 3 mL per minute.

11.5.8 Separate TEVA cartridge from UTEVA cartridge. Place new syringe on the UTEVA cartridge.

NOTE: If Uranium analysis is not requested, the UTEVA cartridge is omitted.

11.5.9 Thorium Elution

11.5.9.1 Place a new clean labeled centrifuge tube in the rack beneath the TEVA cartridge.

11.5.9.2 Add 20 mL of 9M HCl into each cartridge and collect eluant. Adjust flow to approximately 1 mL per minute.

11.5.9.3 Add 5 mL of 6M HCl into each funnel and collect in the same centrifuge tube as in the previous step. This 6M HCl rinse will strip any residual traces of Thorium from the cartridge. Adjust flow to approximately 1 mL per minute.

11.5.9.4 For Thorium analysis:

11.5.9.4.1 Transfer the Thorium HCl solution to a clean labeled beaker (save centrifuge tubes) and evaporate to near dryness (only a few mL should be left in the beaker).

11.5.9.4.2 Add 5-10 mL of 1M HCl to the beaker and let sit for a few minutes to cool.

11.5.9.4.3 Transfer sample back to original centrifuge tube using 1M HCl (final volume should be between 20-25 mL).

11.5.9.4.4 **To coprecipitate the Thorium**, proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.10 Plutonium Elution (**Thorium Elution step must be completed before this step- even if Thorium is not requested)**

11.5.10.1 Place a clean, labeled 50 mL centrifuge tube below each TEVA cartridge.

11.5.10.2 **For samples not suspected of containing Neptunium:**

11.5.10.2.1 **Plutonium Elution:**

11.5.10.2.1.1 Mix the 1M HCl and $TiCl_3$ by adding 10 mL of the 1M HCl to the column and pipetting 0.25 mL of the 10% $TiCl_3$ into the column. (0.125 mL of 20% TiCl3).

11.5.10.2.1.2 Add another 10 mL of 1M HCl.

11.5.10.2.1.3 Adjust the column box so that the flow is approximately 1 mL per minute.

11.5.10.2.2 **Collect the plutonium eluant,** and proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.10.3 **For samples suspected of containing Neptunium:**

11.5.10.3.1 **Plutonium Elution:**

11.5.10.3.1.1 Mix the HCl and $TiCl_3$ by adding 10 mL of 9M HCl to the column and pipetting 0.4 mL of the 10% $TiCl_3$ into the column (0.2 mL of 20% TiCl3).

11.5.10.3.1.2 Add another 10 mL of 9M HCl.

11.5.10.3.1.3 Adjust the column box so that the flow is approximately 1 mL per minute.

11.5.10.3.1.4 **Collect the plutonium eluant.**

11.5.10.3.1.5 **To co-precipitate the Plutonium** proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.11 Uranium Elution (if requested)

11.5.11.1 Place a waste 50 mL centrifuge tube below each UTEVA cartridge.

11.5.11.2 Add 5 mL of 3M HNO_3 into each cartridge, adjust flow to approximately 3 mL per minute.

11.5.11.3 Add 5 mL of 9M HCl into each UTEVA cartridge and allow to drain. Adjust the flow rate to 1mL per/minute.

11.5.11.4 Discard this rinse.

NOTE: This rinse converts the resin to the chloride system. Some Neptunium may be removed here.

11.5.12 Add 20 mL of 5M HCl- 0.05M oxalic acid into each cartridge. Adjust flow rate to 1 mL per/minute and allow to drain. Discard this rinse to acid waste.
Note: This rinse removes neptunium and thorium from the cartridge. The 9M HCl and the oxalic acid remove any residual ferrous ion.

11.5.13 Ensure that clean, labeled tubes are placed in the tube rack under the appropriate cartridge.

11.5.14 Add 15 mL of 1M HCl into each cartridge to strip the **Uranium**. Adjust flow rate to 1mL per/minute and allow to drain.

11.5.15 **To co-precipitate the Uranium**, proceed to ST-RC-0100, "Actinide Coprecipitation."

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Density: Sample weight divided by the volume of said sample weight.

12.1.1 D=M/V D=Density, M=Mass and V=Volume

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 Data assessment does not pertain to this sample preparation procedure.

13.1 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.

14.2 Demonstration of Capability

1.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

1.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

1.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and

significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Eichrom Technologies, Inc Analytical Procedure "ACW13 VBS Thorium, Plutonium and Uranium in Water (with Vacuum Box System". January 2003

17.2 Eichrom Technologies, Inc Analytical Procedure "ACW01 Uranium and Thorium, in Water". April, 2001

17.3 HASL-300 EML Procedures Manual 28th Edition "U-02-RC Isotopic Uranium in Biological and Environmental Materials", February 2000

17.4 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.5 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.6 Associated SOPs (current revisions)

17.6.1 ST-PM-0002, Sample Receipt and Chain of Custody
17.6.2 ST-QA-0002, Standards and Reagent Preparation
17.6.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.6.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.6.5 ST-RC-0004, Preparation of Soil , Filter, Biota and Oil & Grease Samples for Actinide Analyses.
17.6.6 ST-RC-0100, Actinide Coprecipitation
17.6.7 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.6.8 ST-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)

18.0 CLARIFICATIONS AND MODIFICATIONS TO REFERENCED METHODS

18.1 Ascorbic acid is used in place of ferrous sulfate to do the Plutonium valance adjustment.

18.1.1 We use ascorbic acid which provides the oxidation without introducing any iron, which is a known interference.

18.2 A 20 mL rinse is done instead of a 5 mL to ensure that all Uranium is rinsed from the cartridge.

18.3 The rinse steps are eliminated in the Plutonium separation step due to the larger rinse prior to the separation of the cartridge.

18.4 Plutonium is eluted with 20 mL 1M HCL to 25 mL $TiCl_3$ which serves to oxidize the Plutonium and strip it from the column in place of 25 mL of 0.05M HNO3/0.05M HF/0.02M $TiCl_3$.

18.5 Bromocresol purple indicator is used throughout, where as the Eichrom method switches from Bromocresol purple to phenolphthalien indicator in mid-process.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Updated to TestAmerica format.
19.2 Updated standards and reagents in section 7.0 and removed Sulfuric Acid from list.
19.3 Updated procedure in section 11.0 regarding centrifuge time length and removed "Pipet" replacing it with "Add" throughout the section.
19.4 Rev. 13:
- 19.4.1 Updated the section 11.4 in reference to the amount of time sample must remain in centrifuge.

19.5 Rev. 14:
- 19.5.1 Added Hydrochloric Acid to Cresol Red indicator solution in section 7.9.
- 19.5.2 Added a note to section 11.5.1.1.3 stipulating that samples with 5mL or more of gel/sediment need to go though a clean-up process.
- 19.5.3 Added section 11.6 for Actinide Extraction from Gel/Sediment formation.

19.6 Rev. 15:
- 19.6.1 Updated the summary of method in section 2.0.
- 19.6.2 Updated equipment used in section 6.0.
- 19.6.3 Updated reagents & standards throughout section 7.0.
- 19.6.4 Removed the use of Cresol Red Indicator solution from section 7.0.
- 19.6.5 Updated section 8.0 regarding storage, collection, pH testing and preservatives of samples.
- 19.6.6 Added uranium to the list of analytes used for laboratory control samples in section 9.3.
- 19.6.7 Updated section 11.0 regarding water sample prep, calcium phosphate precipitation and thorium/plutonium/uranium separation using Eichrom resins.
- 19.6.8 Removed instruction for actinide extraction from gel/sediment formation in section 11.0.
- 19.6.9 Updated calculation for sample density in section 12.0

19.7 Rev. 16:
- 19.7.1 Section 7, removed reference to NIST traceable
- 19.7.2 Section 8, removed holding time requirement
- 19.7.3 Section 9, MB composition updated and LCS requirements updated.
- 19.7.4 Section 11.5.5 updated
- 19.7.5 Section 15 updated

19.8 Rev. 17:
- 19.8.1 Grammatical Errors fixed through out SOP
- 19.8.2 Updated Section 7.0 – updated Nitric acid make up in Section 7.13
- 19.8.3 Updated Section 11.0 – added clarifications to procedure steps
- 19.8.4 Updated Section 17.0 – update title of ST-RC-0004

19.9 Annual Review, no changes (06/15/2015)
19.10 Annual Review, No Changes (05/23/2016)
19.11 Revision 18: (5/30/17)
- 19.11.1 Updated section 5.0
- 19.11.2 Updated section 7.0
- 19.11.3 Updated section 11.0
- 19.11.4 Fixed grammatical errors through out SOP.

19.12 Revision 19: (07/21/17)

19.12.1 Updated section 11 to include sodium nitrite pre-treatment.
19.12.2 (02/27/18) Added references to HASL-300 in section 1 and section 17. No procedural changes.

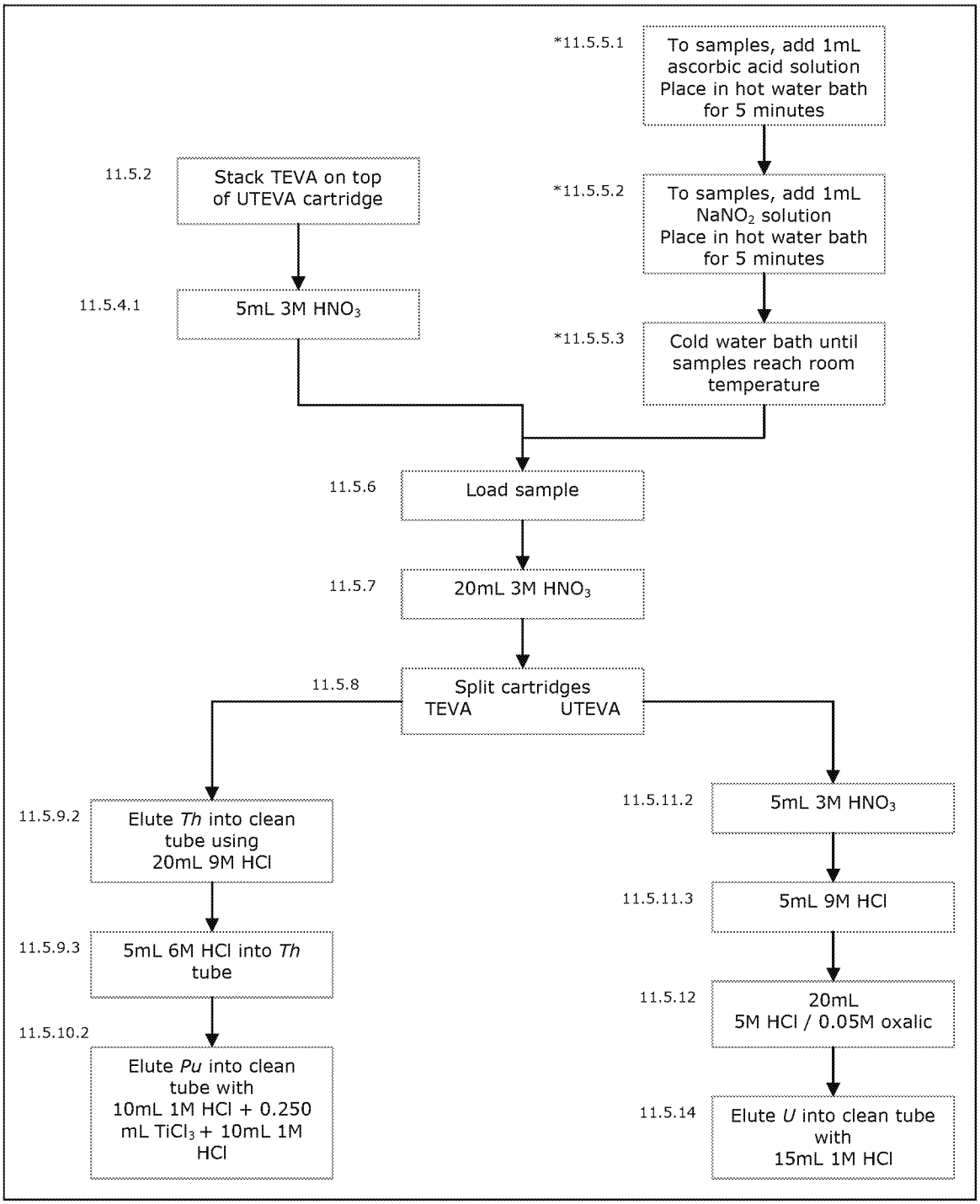
19.13 Revision 20: (06/15/2018)
19.13.1 Technical Review: S. Bernsen/QA Review M Ward
19.13.2 Updated section 7.0 – added 20% TiCl3
19.13.3 Removed ammonium thiocyanate crystals
19.13.4 Updated section 11.0- removed any reference to sodium nitrite pre-treatment
19.13.5 Clarified instructions on adjusting column boxes
19.13.6 Removed the ammonium thiocyanate adjustment
19.13.7 added 20% TiCl3 and aliquots used.

19.14 Revision 21: (08/22/2018)
19.14.1 Technical Review: S. Bernsen
19.14.2 Updated section 11.5.2: Added, for batches that are being performed sequentially with stacked cartridges, the LCS for the element taken from the lower cartridge should be treated the same as the samples. Ex: the LCS for a uranium sample, being run sequentially with thorium, will be loaded through a TEVA/UTEVA stacked cartridge. For single analyte/element analyses, cartridges that are below the cartridge used to bind the analyte of interest, are NOT required to be utilized. Ex: For a plutonium/thorium LCS, the UTEVA cartridge would be omitted.

19.15 Revision 22 (09/23/2019) Technical Review – C. Mazariegos, QA Review – K. Ely
19.15.1 Added Eurofins logo, updated copyright information.
19.15.2 Removed instructions on filtering water samples; filtration should be performed only if requested and following ST-IP-0015.
19.15.3 Corrected acronym for sample duplicate (replaced "SD" with "DU")

Sequential Thorium, Plutonium and Uranium via TEVA/UTEVA

All rinses should flow at 1mL/minute (only 3M HNO_3 may be done at 3mL/minute)
*only necessary when analyzing for *Plutonium*



Title: GAMMAVISION® ANALYSIS

Approvals (Signature/Date):

Jody Watson 11-8-19 Jody Watson Date Radiochemistry Count Room Manager	Michael Ridenhower 11/11/19 Michael Ridenhower Date Health & Safety Manager / Coordinator
Kristen Ely 11/11/19 Kristen Ely Date Quality Assurance Manager	Sarah Bernsen 11/8/19 Sarah Bernsen Date Radiochemistry Operations Manager

This SOP was previously identified as SOP No. ST-RD-0102 Rev. 18

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1.0 SCOPE AND APPLICATION

1.1 This procedure applies to all germanium detectors and the computer assisted germanium spectroscopy analysis system.

1.2 Due to the nature of gamma spectroscopy, once the system is calibrated to a particular geometry a similar matrix may be run as long as it is prepared to match a calibrated geometry.

1.3 This SOP is based on EPA Method 901.1, DOE EML HASL 300 Method GA-01-R and ANSI N42.14-1999.

1.4 The requested limits (**RL**), minimum detectable amount (**MDA**) and QC limits are maintained in the Laboratory Information Management System (**LIMS**).

2.0 SUMMARY OF METHOD

2.1 This procedure provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the germanium spectroscopy system.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Germanium spectrometry has potential interference. Interferences are usually in the form of radionuclides with unresolved photon emissions. These interferences are limited by the careful design/construction of the gamma spectral identification and interference libraries.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.0 EQUIPMENT AND SUPPLIES

6.1 Germanium spectroscopy system utilizing a computer based data acquisition system (GammaVision®-32).

6.2 GammaVision®-32 (know as GammaVision) is a comprehensive, all-in-one package, for the analysis of gamma-ray spectra acquired with HPGe detectors.

6.3 Global Value software is an optimization tool for automation, custom reporting, quality assurance and data management (GammaVision productivity add-on software).

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002. Samples may be collected in glass or plastic containers.

8.2 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 See gamma preparation SOP (ST-RC-0025) for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample, and Sample Duplicate.

9.3 **Method Blank (MB)**

9.3.1 A method blank must be counted with every sample batch.

9.3.2 The method blank is in the same geometry as the majority of the samples in the batch.

9.3.3 The method blank is a pre-prepared empty geometry that is analyzed with every batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 A LCS must be counted with every sample batch.

9.4.2 The LCS is in the same geometry as the majority of the samples in the batch

9.4.3 An LCS is a blank matrix spiked with a known amount of analyte(s).

9.4.4 The LCS is either a purchased sealed source standard or is made in-house.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is a recounted field sample to demonstrate instrument precision, since there is no sample preparation (required to count on a different detector than the sample).

9.5.1.1 If requested, the laboratory may perform a Sample Duplicate which is an additional aliquot of a field sample.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Except in specific instances, it is NOT acceptable to remove points from a calibration curve. Points must never be removed solely for the purpose of meeting criteria. There must be a demonstrable reason to remove a point. Consult with the Supervisor or Technical Director for guidance. Examples of acceptable reasons for the removal of points include, but are not limited to:

10.1.1 A nuclide has decayed below acceptable count criteria due to short half-life

10.1.2 There is an apparent nuclide-specific issue associated with the purchased standard (e.g. all energy lines for a particular nuclide are low or high).

10.2 There are two types of Calibrations performed for Gamma: Energy and Efficiency

10.2.1 Energy Calibrations

10.2.1.1 Frequency: the energy calibration is performed once per detector . The source is not geometry specific.

10.2.1.2 A new calibration curve must be generated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in instrument response, or when indicated by corrective actions.

10.2.1.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to Section 10.1.

10.2.1.4 Range: the energy range is determined by the sample matrix (e.g. 46.54 to 1836.1 keV).

10.2.1.5 Criteria:

10.2.1.5.1 The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration.

10.2.1.5.2 The energy difference (delta Δ) should be within 0.05% for all calibration points or within 0.2 keV for the calibration points.

10.2.1.5.3 The FWHM must be less than 3.0 keV at 1332 keV.

10.2.1.5.4 FWHM difference (delta Δ) should be within 8% for all calibration points.

10.2.2 Efficiency Calibrations

10.2.2.1 Frequency: the efficiency calibration is performed per geometry.

10.2.2.2 A new calibration curve must be generated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in instrument response, or when indicated by corrective actions.

10.2.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to Section 10.1.

10.2.2.4 Range: the energy range of the calibration is determined by the sample matrix e.g.. 46.54 to 1836.1 keV.

10.2.2.5 Broad-Range Energy/Efficiency Curve Criteria:

10.2.2.5.1 The curve should have at least eight points to determine the efficiency.

10.2.2.5.2 The calibration source must have radionuclides that "bracket" the intended range of calibration.

10.2.2.5.3 A minimum of 10,000 counts will be accumulated for each data point.

10.2.2.5.4 The efficiency difference (delta Δ) should be within 8% of the true value for each point, except when utilizing the True Coincidence Correction (TCC) calibration. In the TCC calibration, the nuclides subjected to significant coincidence

events (e.g. Cs-134, Y-88) will appear to be biased low and the lowest energy point (e.g. Pb-210 at 46.5 keV) may have greater variance. This is to be expected, and the apparent bias is corrected in the samples for these nuclides through the TCC correction function of the software. After reprocessing the calibration count using the new TCC calibration, all points should fall within 8% of the true/expected value for each point.

10.3 Initial Calibration Verification (ICV) [Frequency: Once]

10.3.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.3.1.1 The ICV check does not include short half-life nuclides which may exist in the purchased standard. At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).

10.3.2 An ICV must be performed with every initial calibration.

10.3.3 The ICV percent recovery must be within ± 10% of the true value for each nuclide.

10.3.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Daily Checks

10.4.1 The detector **background** shall be checked each day that the germanium spectroscopy system is used. Limits are set at 2 sigma and 3 sigma.

10.4.1.1 <u>Bkgd Countrate (background count rate for entire spectrum)</u>

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.4.2 The instrument **Channel**, **Energy**, **FWHM** (resolution) and **Activity Difference** (efficiency) for a detector shall be checked each day the germanium spectroscopy system is used (using a check source that is non-geometry specific).

10.4.2.1 **Channel** – (low and high energy) is monitored for channel alignment. Limits are set around the target Channel.

10.4.2.1.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	± 1 channel
		Control (out)	=	± 2 channels
10.4.2.1.2	QA-1332	<u>High Energy</u>		
		Tolerance (warning)	=	± 2 channels
		Control (out)	=	± 3 channels

10.4.2.2 **Energy** – (low and high energy) is monitored for energy alignment. Limits are set around a target energy.

10.4.2.2.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	± 0.25 keV
		Control (out)	=	± 0.50 keV
10.4.2.2.2	QA-1332	<u>High Energy</u>		
		Tolerance (warning)	=	± 0.5 keV
		Control (out)	=	± 0.75 keV

10.4.2.3 Full-Width at the Half Maximum (**FWHM**) - (low, mid, and high energy) is monitored for peak shape There are no limits compared to a target FWHM. There are no lower limits (–) set for FWHM.

10.4.2.3.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	+ 1.1
		Control (out)	=	+ 1.2
10.4.2.3.2	QA-662	<u>Mid Energy</u>		
		Tolerance (warning)	=	+ 1.7
		Control (out)	=	+ 1.8

10.4.2.3.3 QA-1332 High Energy

Tolerance (warning)	=	+ 2.2
Control (out)	=	+ 2.3

10.4.2.4 **Activity Difference** (low, mid, and high energy) – is monitored to check the percent difference between the source activity and the reported activity. Limits are set around the target activity.

10.4.2.4.1 QA-60/662/1332 Low/Mid/High Energy

Tolerance (warning)	=	± 4
Control (out)	=	± 5

10.4.3 If the daily check is outside of the control limits, it may be recounted (QSM and other project-specific quality plans may require two consecutive passing source counts before the detector can be used to count project samples).

10.4.3.1 If the out of control parameter is found acceptable for the rerun(s), the instrument can be used for the analysis of samples. ***Note***: *No corrective action is necessary for this situation since the uncertainty can be attributed to the stochastic uncertainty of decay process (statistics), uncertainty of the sources, or a known uncorrected trend.*

10.4.3.2 If the instrument fails to meet the acceptance criteria for the rerun, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out". (See ST-QA-0036 for NCM details regarding tagging out of service).

10.4.3.3 If the QC check fails the following day for the same detector for the same specific parameter as the day before, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out" until the detector can be evaluated and/or maintenance can be performed.

10.4.3.4 The analyst may want to:

10.4.3.4.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.

10.4.3.4.2 Check source positioning and all instrument settings.

10.4.3.4.3 Check all cables for any apparent damage and confirm that all cables are routed to proper connectors and are in good working order.

10.4.3.4.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Corrective actions must be noted in the instrument maintenance log.

10.4.3.4.5 If a parameter has two successive values in the warning limits, the system will be examined for a trend and noted in the maintenance log. Decisions will be based upon the Data Quality Objectives (DQO) and the degree of the bias in relation to the parameter.

10.5 Background

10.5.1 Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements.

10.5.1.1 Backgrounds count for a minimum of 12 hours.

10.5.1.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.

10.5.1.2 Monthly Background limits are set at 2 sigma and 3 sigma.

10.5.1.2.1 Bkgd Countrate (background count rate for entire spectrum)

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.6 Calibration Software Handling

10.6.1 Gamma Detector System Energy and Shape Calibration

10.6.1.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.1.1.1 X = Detector Number
10.6.1.1.2 Analysis method
10.6.1.1.3 Select library
10.6.1.1.4 Enter correct sample data.
10.6.1.1.5 Enter correct conversion time.

10.6.1.2 Close all detectors windows in the current instance of gamma vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.1.3 Select the menu "Analyze\Setting\Sample type…."

10.6.1.4 Select the browse button next to the "File" field and open the file. Click the "OK" button of the window to close it.

10.6.1.5 Recall the application Calibration File from the menu "Calibration \Recall Calibration…"

10.6.1.6 Select the menu "Calibrate\Calibration wizard…"

10.6.1.7 Select the option to create new energy calibrations. Select the next button.

10.6.1.8 On the energy calibration wizard page, select the file "DET_EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the next button.

10.6.1.9 Select the next button to perform the energy, FWHM.

10.6.1.10 Select the edit energy button to review the energy.

10.6.1.10.1 Close the energy calibration sidebar window.

10.6.1.11 Select the save calibration button and save the calibration to "Cal\Energy\X_Energy.clb" where X is the detector.

10.6.1.12 Enter the calibration description in the format "X_ENERGY_GEOMETRY" where X is the detector number and Geometry is an appropriate geometry description when prompted. Select the Finish button to close the calibration wizard.

10.6.1.13 Print the calibration report from the menu "Calibrate \print calibration.

10.6.2 Gamma Detector System Efficiency Calibration

10.6.2.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.2.1.1 X = Detector Number
10.6.2.1.2 Analysis method
10.6.2.1.3 Select library
10.6.2.1.4 Enter correct sample data.
10.6.2.1.5 Enter correct conversion time.

10.6.2.2 Close all detector windows in the current instance of Gamma Vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.2.3 Select the menu "Analyze\Setting\Sample Type".

10.6.2.4 Select the browse button next to the "File", field and open the file. Click the "OK" button at the bottom of the window to close it.

10.6.2.5 Recall the applicable calibration file from the menu "Calibration\Recall Calibration" (if the geometry file currently exists).

10.6.2.6 Select the menu "Calibrate\Calibration Wizard".

10.6.2.7 Select the option to create new energy and efficiency calibration. Select next button.

10.6.2.8 On the Energy Calibration Wizard page select the file "EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the Next button.

10.6.2.9 On the Efficiency Calibration Wizard page, select library file, "DET_EfficiencyCalibration.Lib" for mixed gamma sources.

10.6.2.10 On the Efficiency Calibration Wizard page, select the appropriate Certification file from the directory.

10.6.2.11 Select the next button to perform the energy FWHM and efficiency calibration.

10.6.2.12 Select the Edit Energy button to review the energy and FWHM Calibration.

10.6.2.12.1 Close the Efficiency Calibration side window.

10.6.2.13 Select the save calibration button and save the calibration to Cal\X_Geometry.clb" where X is the detector and geometry is an appropriate geometry name.

10.6.2.14 Enter the calibration description in the format "x_Geometry_Source number_date counted" where X is the detector number and geometry is an appropriate geometry description when prompted. Select the finish button to close the calibration wizard.

10.6.2.15 Print calibration report from the menu "Calibrate\Print Calibration".

10.6.2.16 Select "Analyze", select "Entire spectrum in memory" and file print.

10.6.2.17 Close the spectrum Buffer window and save the spectrum when prompted.

10.6.3 Detector Long Background Counting

10.6.3.1 Remove any samples from the detector, clean the detector, close the shield lid and start acquisition.

10.6.3.2 Select detector in global value quick Start.

10.6.3.3 Select Monthly Background PBC under Automation Groups.

10.6.3.4 Select Background PBC Long Count under Automation Jobs.

10.6.3.5 Login using name and password.

10.6.3.6 Select "OK", ensure detector cave is empty.

10.6.3.7 Repeat for each detector which background you would like to start.

10.6.3.8 After the background is complete it will save as a PBC file.

10.7 Evaluation of Controls for Trends

10.7.1 Identified controls shall be evaluated for trends at a minimum frequency of 1 month, but as often as deemed by analysts. The controls and trending rules are listed below (see appendix for list of trending rules).

10.7.1.1 Background FWHM, Centroid (by engergy), Efficiency

10.7.1.1.1 Trending Rules 2, 3 & 6

10.7.1.2 Source FWHM, Centroid (by engergy), Efficiency

10.7.1.2.1 Trending Rules 2, 3 & 6

11.0 PROCEDURE

11.1 Calibration Quality Control (**Daily Check**)

11.1.1 Place the calibration quality control sample on the detector, and start acquisition.

11.1.2 Select detector from global value quick start.

11.1.3 Select Quality Control under Automation Groups.

11.1.4 Select Daily Quality Control Check under Automation Jobs.

11.1.5 Login with user name and password.

11.1.6 Select "OK", ensure source is on detector.

11.1.7 Repeat for each detector.

11.1.8 Record in the instrument run log.

11.2 Background Quality Control (**Daily Background**)

11.2.1 Remove any samples from the detector, and start acquisition.

11.2.2 Select detector global value quick start.

11.2.3 Select quality control under automation groups.

11.2.4 Select daily background check under automation jobs.

11.2.5 Login with username and password.

11.2.6 Select "OK", ensure detector cave is empty.
11.2.7 Repeat for each detector.
11.2.8 Record in the instrument run log.

11.3 Sample Counting
11.3.1 Place the sample on the detector.
11.3.2 Select detector from global value quick start.
11.3.3 Select analyze samples under automation groups.
11.3.4 Select count sample under automation jobs.
11.3.5 Login with username and password.
11.3.6 Scan sample description from barcode report.
11.3.7 Select analysis method, sample type, geometry, library, correct date, count time, and continue.
11.3.8 Select "OK", ensure sample is on detector.
11.3.9 Record in the instrument run log.

11.4 Analysis/Analyte Specific Considerations
11.4.1 Analysis library
11.4.1.1 The analysis library, if supplied by the laboratory, should use nuclide energy lines and abundances from the table of isotopes edited by Gerhard Erdtmann and Werner Soyka.
11.4.1.2 The analysis library used should be representative of the types of analytes expected to be seen in the project samples.
11.4.1.3 The analysis library may be supplied by the client, defining their project-specific analytes, assumptions, energy lines, and abundances.
11.4.2 Ra-226 analysis using the nuclide's 185.99 keV peak
11.4.2.1 Ra-226 may be analyzed using the 3.28% abundant peak at 185.99 keV. This analysis may be performed using any geometry any time after preparation of the sample (including "0-day"). Ra-226 as a nuclide must be included in the analysis library with the abundance and energy listed.
11.4.2.1.1 Note: The 54% abundant 185.72 keV gamma ray emission of U-235 may produce a significant interference. This can produce high-bias to Ra-226 due to counts from the higher-abundant U-235 peak being mis-classified into the Ra-226 peak. However, there are circumstances where the opposite may occur (Ra-226 counts "lost" into the U-235 peak), producing a low bias. The client should be aware of the potential interferences.
11.4.3 Ra-226 inferred from Bi-214 after 21-days of ingrowth
11.4.3.1 Ra-226 may be inferred from the Bi-214 daughter after ingrowth in a sealed container devoid of headspace. This is the preferred means for determining Ra-226 by gamma spectrometry.
11.4.3.1.1 The ideal ingrowth period is 21-days (or longer), resulting in nearly complete secular equilibrium of the decay chain through Rn-222 down to the Bi-214 progeny. Other client-requested ingrowth periods (e.g. 14-days, 10-days, 7-days) may be used, but a NCM should be written stating the reported Ra-226 result may be low-biased due to the shorter ingrowth period.
11.4.3.1.2 The container should be reasonably well sealed to ensure gaseous Rn-222 does not escape the container.
11.4.3.1.3 The container (geometry) should not have significant headspace, into which Rn-222 could escape, ultimately resulting in a low bias. If such a geometry is used (e.g. due to limited aliquot provided by the client), a NCM should be written.
11.4.4 Other inferences:
11.4.5 Oftentimes nuclides, which may not have any reasonable gamma emissions, are reported based upon either shorter-lived daughters or a longer-lived parent, assuming

secular equilibrium between the two. The half-life used for any decay corrections should be that of the longer-lived parent. Note: Such assumptions should be evaluated by the client as to appropriateness to their sample type. For example, Th-232 is often inferred from the Ra-228 daughter. While this is a good assumption for most soil samples, this is likely not a good assumption for an oil & gas flowback water. Common inferences ("re-maps") include:

input analyte	remapped to analyte
Ac-227	Th-227
Ac-228	Th-232
Ac-228	Ra-228
Ag-108m	Ag-108
Bi-214	Ra-226
Cs-137	Ba-137m
Pb-210	Po-210
Pb-210	Bi-210
Pb-212	Th-228
Pb-212	Ra-224
Rh-106	Ru-106
Sb-125	Te-125m
Th-227	Ra-223
Th-227	Ac-227
Th-227	Bi-211
Th-227	Pb-211
Th-231	U-235
Th-234	Pa-234
Th-234	U-238
U-235	Th-231

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 All calculations are performed in GammaVision-32 software; conversions are performed in RadCapture. Calculations are found in ST-QAM.

12.3 Other Detectable Radionuclides (ODRs - Identified as TICs in LIMS) when requested shall be evaluated when the activity to MDC ratio (Act/MDC) is above two (2) unless specified by the Client Requirement Memo (CRM) or by project notes in LIMS.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (**MB**)

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the requested reporting limit (RL). If no requested limit is assigned to the analyte(s), the activity to MDC ratio (Act/MDC) shall be below two (2). No further action would be required at that point.

13.2.1.2 Project specific requirements, if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the Client Requirement Memo or in project notes in LIMS..

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the method blank exhibits activity above the limits/criteria (see Section 13.2.1), the affected analytes shall be evaluated to determine possible force fitted peak, or unsupported energy lines using the Gamma Vision™ raw data reports. If the contaminant is found to be unsupported, the analyst will narrate findings in an NCM.The blank may be re-counted once to confirm the activity. The recount can be used to report only the affected analyte(s); the original count can be used to report all unaffected analyte(s) if the analyst finds no additional error in the original count. If the re-counted MB activity exceeds the criteria/limit further evaluation will be performed and narrated accordingly. Note that certain analytes are common laboratory contaminants which require special narration.

13.3 Laboratory Control Sample (**LCS**)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – The LCS may be re-counted once to confirm the result. If the re-counted LCS exceeds the control limit, samples that are non-detect may be reported with an NCM.

13.3.2.2 LCS Spike Recovery excursion (low) – The LCS may be re-counted once to confirm the result. If the low recovery is confirmed, the batch is recounted.

13.4 Duplicate

13.4.1 Acceptance Criteria:

13.4.1.1 All control analytes must be within the specified control limits for precision (RPD), max. 40% RPD, RER < 1, and/or DER ≤ 3.

13.4.2 Corrective Action for Duplicate not meeting acceptance criteria:

13.4.2.1 RPD/RER Duplicate excursion – The sample is recounted if both RPD and RER (and/or DER) exceed criteria.

13.5 Insufficient Sample

13.5.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative. The insufficient sample description is included in the LIMS NCM within the type defining the excursion.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are documented in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration of capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 28th Edition, method GA-01-R, Gamma Radioassay

17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1

17.3 American National Standards Institute (ANSI) Accredited Standards Committee on Radiation Instrumentation, N42; ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides

17.4 Ortec MCB Connections-32, Hardware Property Dialogs Manual, current version

17.5 MAESTRO–32, MCA Emulator, current version

17.6 GammaVision–32, Gamma-Ray Spectrum Analysis and MCA Emulator, current version

17.7 Master library Source: Gerhard Erdtmann, Werner Soyka

17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.11 Associated SOPs, Current Revision:

17.11.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples

17.11.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis

17.11.3 ST-RC-0025, Preparation of Samples for Gamma Spectroscopy

17.11.4 ST-QA-0002, Standards and Reagent Preparation
17.11.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.11.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.
19.2 Revision 8:
- 19.2.1 Increased background count times from 12 to 36 hours in section 10.3.1.1.
- 19.2.2 Updated the procedure for detector long background counting in section 10.5 to reflect new software.
- 19.2.3 Updated daily calibration checks, daily background and sample counting procedures in section 11.0 to reflect new software.

19.3 Revision 9:
- 19.3.1 Replaced quartz sand with sodium sulfate to be used for soil method blanks in section 9.2.
- 19.3.2 Updated section 10.4 regarding instrument daily checks.
- 19.3.3 Updated data assessment and acceptance criteria in section 13.0
- 19.3.4 Updated section 9.0 regarding batch, method blank and laboratory control samples.
- 19.3.5 Updated the calibation points for an internal calibation in section 10.1.
- 19.3.6 Updated the percent recovery regarding the ICV in section 10.2.
- 19.3.7 Updated software storage file name throughout section 10.5.

19.4 Revision 10:
- 19.4.1 Updated references to QuantIMS through out
- 19.4.2 Update §10.1
- 19.4.3 Added §10.3 Annual Calibration Verification
- 19.4.4 Updated §10.4: 36 hour background changed to 12 hour and requirement to complete Attachment 2
- 19.4.5 Added Attachment 2, "Monthly Background Complete" example
- 19.4.6 Updated §13 references to Clouseau changed to LIMS
- 19.4.7 Added §17 reference to ANSI 42.14-1999

19.5 Revision 11:
- 19.5.1 Updated §1.4 with corrected termonolgy
- 19.5.2 Updated §6.0 software details
- 19.5.3 Additon/Update §10.0 major change in calibration
- 19.5.4 Updated §13.0 additional corrective action steps
- 19.5.5 Updated §15.0 with new verbiage

19.6 Revision 12: (04/16/2014)
- 19.6.1 Spelling and grammar corrections made throughout SOP.
- 19.6.2 Sections 10.2.3 and 10.2.5 had wording changed to common text.
- 19.6.3 Section 10.4.3.3 was updated to add 'for the same specific parameter as the day before' and 'until the detector can be evaluated and/or maintenance can be performed.'.
- 19.6.4 Section 10.5.1.2.1 was added to provide limits for monthly backgrounds, which were not previously provided.
- 19.6.5 Section 13.4.2 had 'LCS' changed to 'duplicate' since it is the duplicate section and LCS was incorrectly referenced.

19.7 Revision 13: (06/22/2015)
- 19.7.1 Section 10.5.1.1 was updated to say "Backgrounds count for a minimum of 12 hours" new wording.

19.7.2 Section 10.6.3.2 updated captialzation "global value quick".
19.7.3 Section 11.1.2 updated capitalization "global value quick start".
19.7.4 Section 111.3.2 updated capitalzation "global value".
19.7.5 Section 11.3.7 updated to say "and continue." new wording.

19.8 Revision 14: (05/23/2016)
19.8.1 Trending evaulation added to section 10
19.8.2 Appendix with trending rules added

19.9 Revision 15 (5/2/2017)
19.9.1 True Coincidence Correction (TCC) calibration.onformation added to Section 10.1
19.9.2 Trending Rules changed from 2,3,and 5 to 2,3,and 6

19.10 Revision 16 (12/12/2017) Technical Review: R. Mueller/QA Review: M Ward)
19.10.1 Section 10.1.2.55 changed/edited
19.10.2 Section 10.3 removed ACV mention; ACV is no longer performed

19.11 Revision 17 (3/9/2018) Technical Review: T. Romanko/Rachel Mueller / QA Review: M. Ward
19.11.1 Section 10.4.1.1.2 removed no longer utilized
19.11.2 Added Section 11.4: Analysis/Analyte Specific Considerations
19.11.3 Section 9.1 – fixed SOP reference
19.11.4 Added Section 10.1
19.11.5 Section 10.5.1.1.2 removed
19.11.6 Attachment 2 removed (Monthly Background Complete form); no longer posted
19.11.7 Added Section 12.3 – ODR information
19.11.8 Section 13.2 – added Method Blank criteria

19.12 Revision 18 (3/22/2019) Technical Review: R. Mueller/QA Review M. Ward
19.12.1 Section 10.2 – Added verbage regarding calibration frequency
19.12.2 Section 10.2 – reference QSM daily check requirements- two pass criteria.
19.12.3 Section 13.4 – added DER to precision criteria

19.13 Added Eurofins logo and updated copyright information (4/18/2019)

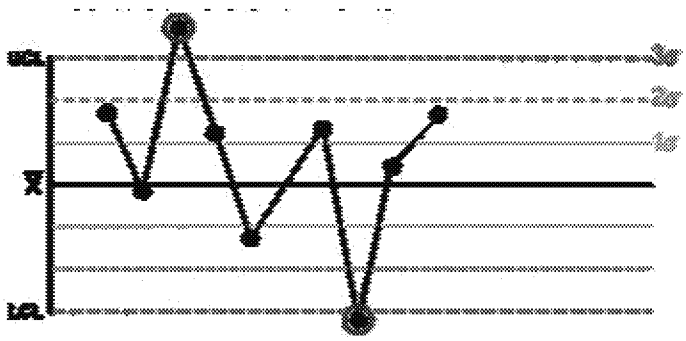
19.14 Revision 19 (11/12/2019) Technical Review – J. Watson ; QA Review – K. Ely
19.14.1 Section 9
19.14.1.1 Updated MB and LCS to match current practice.
19.14.1.2 Removed duplicated paragraph on NCM's.

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.

~ Simplest: Single point is considered out of control event

- Corrective action required



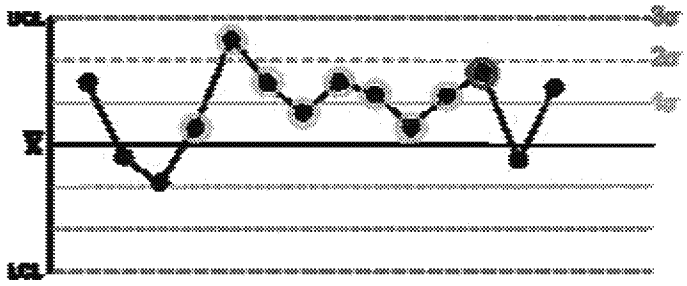
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged bias exists.

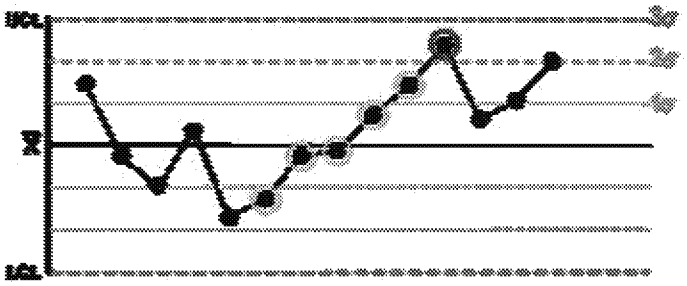
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A trend exists.

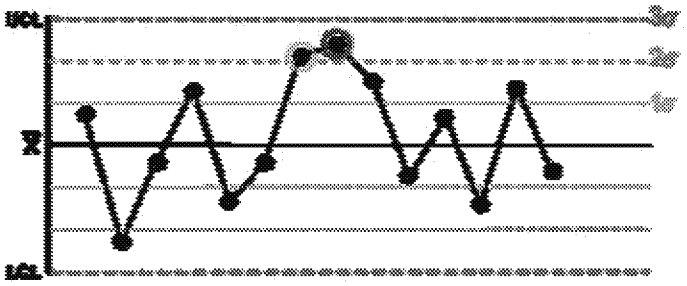
  ~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

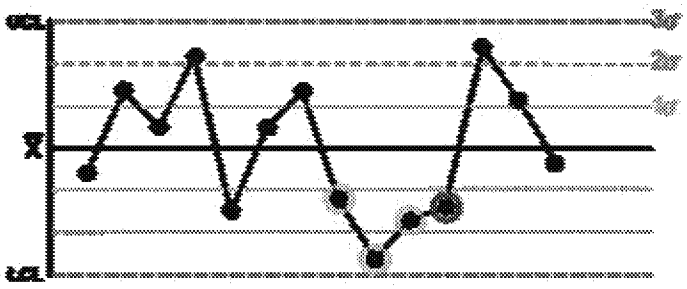
  ~ Investigate for preventive. May not be CAR



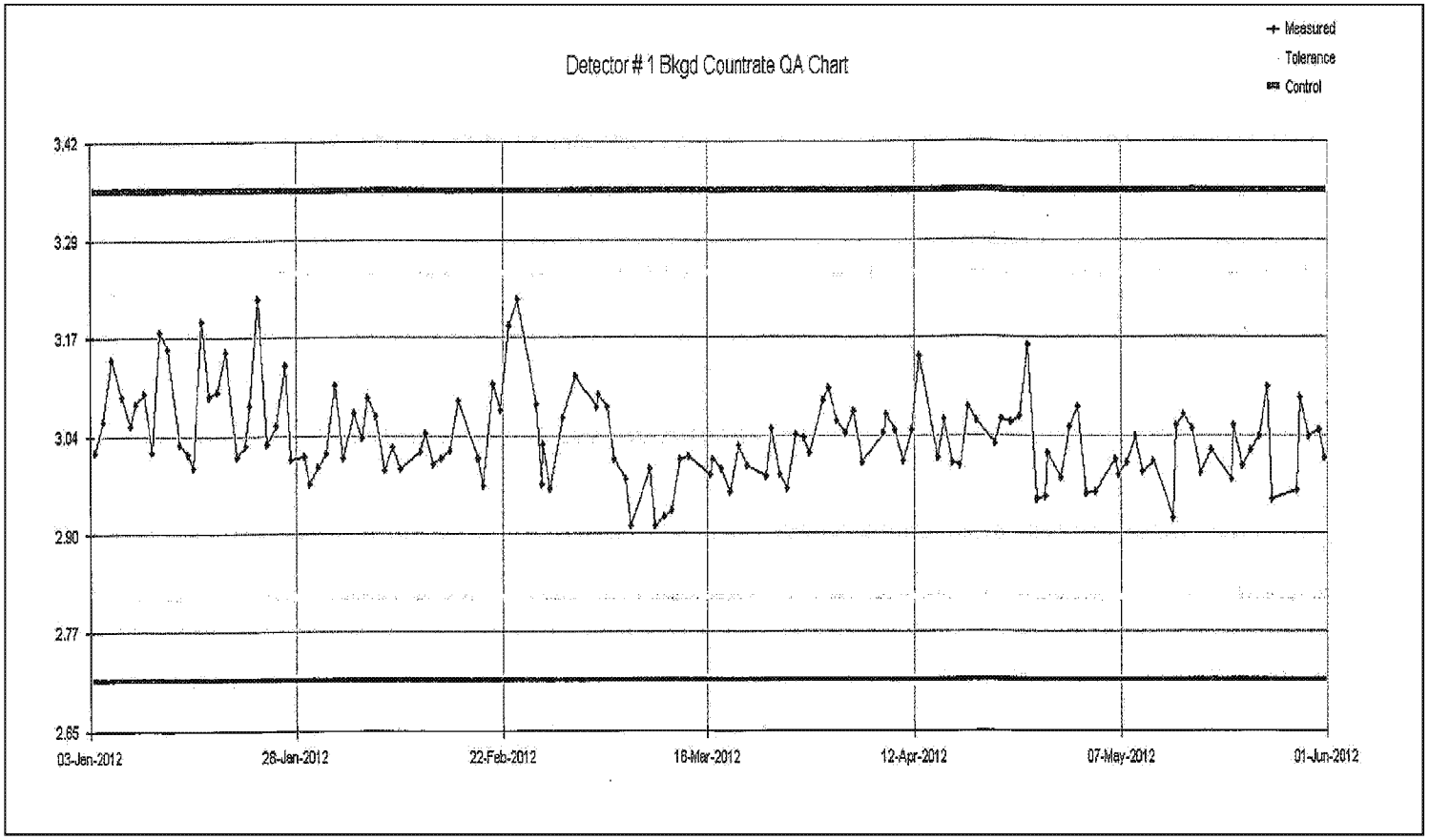
Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

  ~ No CAR, Statistical trend



Attachment 1



Title: ALPHA SPECTROSCOPY ANALYSIS [DOE HASL-300 A-01-R]

Approvals (Signature/Date):

Jody Watson — 9-4-19 — Date Radiochemistry Count Room Manager	Michael Ridenhower — 9/5/19 — Date Health & Safety Manager / Coordinator
Kristen Ely — 9/5/19 — Date Quality Assurance Manager	Jodie Carnes — 9/5/19 — Date Laboratory Director Rad Ops Manager

This SOP was previously identified as SOP No. ST-RD-0210 Rev. 15

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1.0 SCOPE AND APPLICATION

1.1 This procedure applies to alpha spectroscopy detectors and the computer assisted alpha spectroscopy analysis systems, using AlphaVision software.

1.2 This SOP is based on DOE method A-01-R

1.3 This SOP is applicable to both liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the alpha spectroscopy system.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for glossary of common terms and data qualifiers.

3.2 Tracer – A known amount of ^{232}U, ^{242}Pu or ^{236}Pu, ^{243}Am, ^{209}Po, ^{244}Cm or ^{229}Th (depending on analyte(s) required) added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

3.3 Region of Interest (ROI) – The keV range through which the target isotope peak signal responds.

3.4 keV - (kilo electron Volt) – electron volt is a unit of energy defined as the amount of energy gained (or lost) by the charge of a single electron moved across an electric potential difference of one volt.

3.5 Tailing – Tailing is a delayed return of a peak to chromatographic baseline or continuation of response beyond its normal response window (RT window, ROI) due to high concentration of the analyte or matrix interference.

3.6 AlphaVision – The Alpha Spectrometer data collection and processing software.

4.0 INTERFERENCES

4.1 Alpha spectrometry has many potential interferences. These are usually in the form of radionuclides with unresolved alpha emissions. Poorly resolved alpha peaks are often due to high alpha activity rates or attenuation of the alpha emissions.

4.2 Isotope peak responses, when sufficiently high, may tail into other isotope ROI. Th-229 tailing into the Th-230 region of interest is a recognized example. This interference is minimized by maintaining low activities of the Th-229 tracer and monitoring of the separation of the ROI for Th-229 and Th-230. The use of manual integration may be required.

4.3 Some isotopic elements are not distinguishable and are reported as an isotopic pair, unless specifically directed by the client not to do so. These pairs may be reported separately depending

on the client's DQO and the use-ability of the data. When reported separately, the narrative must describe the technical aspects of how the isotopic pair was divided.

4.3.1 Recognized Isotopic Pairs:

4.3.1.1 Plutonium-239/240
4.3.1.2 Uranium-235/236
4.3.1.3 Uranium-233/234
4.3.1.4 Curium-245/246
4.3.1.5 Curium-247/248

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED
None.

6.0 EQUIPMENT AND SUPPLIES

6.1 Alpha spectroscopy system utilizing a computer based data acquisition system.

6.1.1 Hardware – Octete Plus and Alpha Ensemble
6.1.2 Software – AlphaVision and Radcapture

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Commercially prepared alpha standards for the isotopes Th-230, Pu-239 and Am-243.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and the same reagent lot(s).
9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.
9.2.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, a LCS Duplicate (LCSD) is prepared and analyzed.
9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**
9.3.1 A method blank is a blank matrix processed simultaneously with and under the same conditions as samples through all steps of the procedure.
9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**
9.4.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
9.4.2 A LCS must be prepared with every sample batch.

9.5 **Matrix Spike (MS)**
9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate (Dup)**
9.6.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**
9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.
9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.

10.0 INSTRUMENT SETUP, CALIBRATION, AND STANDARDIZATION

10.1 Initial instrument setup is performed when the instrument is first installed, when a detector or chamber is changed/replaced, when a chamber is returned from the manufacturer after servicing, or other such circumstances. The following steps should be taken to ensure proper setup. Steps may be accomplished either through hardware knobs/potentiometers or through software settings (depending upon the system hardware/software). See the hardware and/or software manual to determine further detailed instructions:
10.1.1 Set the conversion gain to 1024 channels.
10.1.2 Adjust the coarse and fine gain as well as the offset to adjust the location of the three peaks of the alpha source such that the lower energy peak (Th-230 at 4688 keV) falls into channel 176, the mid-energy peak (Pu-239 at 5155 keV) falls into channel 239, and the higher energy peak (Am-241 at 5486 keV) falls into channel 283. Note that this results in 107 channels between the low energy and high energy peaks (about 7.46 keV/channel with offset of approximately 3375 keV). Ensure each peak is within 2 channels of the desired channel before beginning energy and efficiency calibrations.

10.1.2.1 Gain adjustment:

10.1.2.1.1 Turning the knob counter-clockwise decreases the gain (decrease the value in the fine gain for software adjustment), causing the spectrum peaks to move closer to each other (smaller keV/channel slope) and toward the lower energy.

10.1.2.1.2 Turning the knob clockwise increases the gain (increase the value in the fine gain for software adjustment), causing the spectrum peaks to spread apart (larger keV/channel slope) and toward higher energy.

10.1.2.2 Offset adjustment:

10.1.2.2.1 Increasing the offset moves the peak/spectra toward the the left (lower channel number) without altering the keV/channel (slope).

10.1.2.2.2 Decreasing the offset moves the peak/spectra toward the right (higher channel number) without altering the keV/channel (slope).

10.1.3 Adjust the pulser setting such that the pulser peak is centered at about channel 222 (approximately 5 MeV).

10.2 Calibrations

10.2.1 Energy calibrations shall be performed prior to initial use, following repair or replacement of a key detector component when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in the instrument response, or when indicated by corrective actions.

10.2.1.1 Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 MeV.

10.2.1.2 Typical isotopes used are Th-230, Pu-239, and Am-241. The energy vs. channel slope should be less than 15 keV per channel. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Final peak energy positions of all observed isotopes shall be within ± 2.5 channels (~20 keV) of expected channel/energy (see 10.1.2). The actual energy vs. channel and the equation with the slope is not calculated. Setting the peaks to within 2.5 channels of expected will ensure calculations utilizing fixed Regions of Interest (ROI) for each isotope will provide accurate results with minimal need for manual adjustment of ROI.

10.2.1.3 Routine pulser checks and continuing calibration verifications (see below) will help control/monitor for drift.

10.2.2 Efficiency calibrations shall be performed prior to initial use, following repair or replacement of a key detector component when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in the instrument response, or when indicated by corrective actions..

10.2.2.1 A minimum of 3000 net counts must be collected for each peak in the calibration. Calibrated efficiency should fall between 20% and 32%. Values outside this range do not constitute a failure. However, if the calibrated efficiency does fall outside this range, an evaluation of the suitability of the detector for use should be performed and documented.

10.2.3 Initial calibration verifications (ICV) shall be performed utilizing an independent second source following the initial calibration.

10.2.3.1 A minimum of 2000 net counts must be collected for each peak in the ICV. The efficiency of the ICV must fall within 95%-105% of the initial calibration efficiency value. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibration peak in the initial calibration (IC).

10.2.3.2 A second level review will be perfomed before detectors are placed into service and will be noted as acceptable in the electronic monthly maintenance log.

10.3 Continuing Calibration Verification (CCV)

10.3.1 A continuing calibration verification shall be performed on a monthly basis.

10.3.1.1 A minimum of 2000 net counts must be collected for each peak in the CCV. The efficiency should fall within 95%-105% of the calibrated efficiency. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 30 keV of the corresponding calibration peak in the initial calibration (IC).

10.3.1.2 A second level review will be performed before detectors are placed into service and will be noted as accepable in the electronic monthly maintenance log.

10.4 Background subtraction spectrum (Initial Calibration Background or ICB) shall be established for the alpha spectroscopy systems monthly, or when the background quality control check indicates an unacceptable change in the daily background parameters.

10.5 Instrument Contamination Check (ICC) shall be performed weekly at minimum and after counting high activity samples (see section 11.9) . Must be a minimum four hour count duration.

10.6 Daily Checks (Pulsers)

10.6.1 Routine pulser quality control verifications are performed each day of use.

10.6.1.1 The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used. The limits for pulser centroid and pulser energy will be as below:

10.6.1.1.1 Gross counts must be within 5% of the average (20-point minimum) for each detector.

10.6.1.1.2 The peak resolution (FWHM) must fall within 10-20 keV.

10.6.1.1.3 The pulser centroid must fall within ± 5 channels of the average (20-point minimum) for each detector.

10.6.1.1.4 The pulser energy must fall within ± 40 keV of the average (20-point minimum) for each detector.

10.6.2 Routine calibration, background and pulser quality control parameters using the "Boundry" out-of-range test will be found unacceptable if the value is outside parameter tolerance.

10.6.2.1 The routine quality control check should be rerun to determine the statistical significance of the out of control parameter.

10.6.2.2 If the out of control parameter is found acceptable in the rerun, the investigation will be noted in the instrument maintenance log.

10.6.2.3 Check the integrity of the radioactive standard.

10.6.2.4 Check source positioning and all instrument settings.

10.6.2.5 Check all cables for any apparent damage and to confirm that all cables are routed to proper connectors and are in good working order.

10.6.3 If the instrument fails to meet the acceptance criteria, and the corrective actions above do not resolve the problem, the instrument must be "tagged" out of service (OOS) for the day see Attachment 1 for OOS tag example.

10.6.3.1 This is noted on the Alpha Spec Daily report (The report will display FAIL for criteria not met). The physical detector will be marked out of service with the date and initials of the analyst performing the daily check.

10.6.3.2 If a detector fails three consecutive days for the same criteria, the detector will be taken out of service until the problem is resolved. This is done by clicking on the detector in Alphavision. Right click on the detector, select detector properties, check the "out of service" box and fill in the description field briefly explaining the problem. Mark the detector with an OOS tag as a visual indicator of its status.

10.6.3.3 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Note any repairs in the maintenance log.

10.7 Calibration process in the Software

10.7.1 Alpha Detector System Energy and Efficiency Calibration (Initial Calibration- IC)

10.7.1.1 Print out the standards sheet for initial calibration (IC).
10.7.1.1.1 Location: \\STLFS01\Rad\alpha\Calibration_Sources
10.7.1.2 Load sources into the detector.
10.7.1.3 In the AlphaVision software, click on the Calibration icon.
10.7.1.4 Click on the detector to be calibrated.
10.7.1.5 Select Process and then select calibration from the dropdown menu.
10.7.1.5.1 The Calibration Explorer Window will appear.
10.7.1.6 In the General Window: scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.7.1.7 Choose the correct source template.
10.7.1.8 Click next.
10.7.1.9 In the Acquisition window, confirm count time of 140 minutes.
10.7.1.10 Click next.
10.7.1.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1) Make sure the 'Active' box is checked so the calibratoin is put in use immediately after calibration is processed.
10.7.1.12 Click next.
10.7.1.13 In the Report Window, select print on completion.
10.7.1.14 Click finish.
10.7.1.15 When count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, Click Calibrate, click Save.
10.7.1.16 Verify the efficiency is above 20% and below 32%.
10.7.1.17 Repeat for each detector.
10.7.1.18 Record the calibration in the Alpha Maintenance Log.

10.7.2 ICV Procedure

10.7.2.1 Print out the standards sheet for ICV.
10.7.2.1.1 Location: \\STLFS01\Rad\alpha\Calibration_Sources
10.7.2.2 Place the correct source into the detector.
10.7.2.3 In The AlphaVision software, click on the Calibration Icon.
10.7.2.4 Click on the detector to be calibrated.
10.7.2.5 Select Process and then select calibration from the dropdown menu.
10.7.2.6 The Calibration Explorer Window will appear.
10.7.2.7 In the General Window; scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.7.2.8 Choose the correct source template.
10.7.2.9 Click next.
10.7.2.10 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1).
10.7.2.11 Click next.
10.7.2.12 In the Report Window, select print on completion.
10.7.2.13 Click finish.
10.7.2.14 When the count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, click Calibrate and click Save.
10.7.2.15 Verify the efficiency is above 20% and below 32%.
10.7.2.16 Repeat for each detector.
10.7.2.17 Record the ICV in the Alpha Maintenance Log.
10.7.2.18 Open the AlphaVision Access database program in \\stlfs01\RAD\Alpha Tools\AV Tool (2003).mdb.
10.7.2.19 Select frm_QC_main from the form tab.
10.7.2.20 Enter date range.
10.7.2.21 Select system 1 for AlphaVision or system 2 for AlphaVision 1.

10.7.2.22 Select Get Cal Data.
10.7.2.23 Exit.
10.7.2.24 Select Check Ver to run the report and verify the ICV passes criteria.
10.7.2.25 For the Intitial Calibratin (IC), the detectors must have the box checked in the Cal Data window for that specific calibration and the previous year's IC must be unchecked and the 'do not use' box must be checked to ensure the correct calibration is being used.

10.7.3 CCV Procedure

10.7.3.1 Print out standards sheet for CCV on the network.
10.7.3.1.1 Location: \\STLFS01\Rad\alpha\Calibration_Sources
10.7.3.2 Load sources into the detectors.
10.7.3.3 In the AlphaVision software, click on the Calibration Icon.
10.7.3.4 Click on the detector to be calibrated.
10.7.3.5 Select process and then select calibration from the dropdown menu.
10.7.3.5.1 The Calibration Explorer Window will appear.
10.7.3.6 In the General Window; scan the source name;AV(detector #)-date (with year month day format YYYYMMDD).
10.7.3.7 Select correct source template.
10.7.3.8 Click Next.
10.7.3.9 In the Acquisition window, confirm count time of 60 mins.
10.7.3.10 Click Next.
10.7.3.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used and select which shelf the source is on. (This will be shelf 1.)
10.7.3.12 Click next.
10.7.3.13 In the Report Window, select print on completion.
10.7.3.14 Click finish.
10.7.3.15 When the count is complete, the Manual Energy and Efficiency Calibration window will appear. In this window, select Calibration ROI, select Calibrate and Save.
10.7.3.16 Verify the efficiency is above 20% and below 32%.
10.7.3.17 Repeat for each detector.
10.7.3.18 Record the CCV in the Alpha Maintenace Log..
10.7.3.19 Open the AlphaVision Access database program in \\stlfs01\RAD\Alpha Tools\AV Tool (2003).mdb

10.7.3.20 Select frm_QC_Main from the form tab.
10.7.3.21 Enter date range.
10.7.3.22 Select system 1 for AlphaVision or system 2 for AlphaVision 1.
10.7.3.23 Select Get Cal Data.
10.7.3.24 Exit.
10.7.3.25 Select Check Ver to run the report and verify the CCV passes criteria.

10.7.4 Detector Background Process (ICB) (See Section 10.4)

10.7.4.1 Select the Batch Icon.
10.7.4.2 Select backgrounds from the Tool Bar.
10.7.4.3 Select Process.
10.7.4.3.1 This will open the General Window in Batch Wizard.
10.7.4.4 Name the background with month year format; MonthYYYY (e.g. January2015).
10.7.4.5 Select correct template (provided by analyst).
10.7.4.6 Click next.
10.7.4.7 In the Sample Window, add all detector names with the format: ICB;AV(detector)#.

10.7.4.8 Click next.
10.7.4.9 In the Acquisition Window, confirm count time is set at 960 minutes (or as long as the longest sample count time).
10.7.4.10 Click next.
10.7.4.11 In the Analysis Set Up Page, select Background Library and Background ROI, check the Use ROI box.
10.7.4.12 Click next.
10.7.4.13 In the Report Window, select print on completion.
10.7.4.14 Click finish.
10.7.4.15 The Detector Assignment worksheet will appear, assign detectors, and select start now.
10.7.4.16 Record the backgrounds in the instrument maintenance log.
10.7.4.17 The background spectrum will be processed by the software.
10.7.4.18 The detectors are identified by the color listed below corresponsing to the background counts

Counts in Region of Interest (i.e. Th230, Th232, U234, U238, Pu238, Pu239):

- 0-2 counts – Blue – Ultra Low Level
- 0-4 counts – Yellow – Intermediate Low Level
- 0-20 counts – Green – Low Level
- **Blue dot detectors – 0-20 counts- No Thorium samples can count on these detectors- Americium/Curium and Plutonium**

See Attachment 1 Detector Color Key

10.7.4.19 Detectors within each categorywith backgrounds above the counts listed are taken out of service for cleaning. A recount is allowed after the primary failure.
10.7.4.20 Detectors may also be removed from service when there is a visible peak present or at analyst judgment.
10.7.4.21 Backgrounds will be 2nd reviewed before placing into service and a notation of acceptable will be listed in the electronic monthly maintenance log.

10.8 Detector Cleaning (This should be done before starting Backgrounds- ICB)
10.8.1 Clean detector surface with ethanol and a clean cotton ball.
10.8.2 Clean the sample tray and place a clean background planchette on the tray.
10.8.2.1 A passing background count is required before returning the detector to service.

10.9 Nuclide Specific Detectors
10.9.1 Due to recoil from thorium isotopes, certain ROIs may build up background counts affecting isotopes such as Cm-244 and Pu-236. Analyses reporting such isotopes shall not be performed on detectors that count thorium samples (Blue Dot Detectors referenced in attachment 1).

10.10 Standard Verification Procedure
10.10.1 Receive manual batch from prep or a test job/batch logged in by QA through the LIMs.
10.10.2 Count for 960 minutes (make sure batch is set up correctly).
10.10.3 After count, open decay corrector (located in QA_Public\FORMS\RAD) to see if isotope you are verifying needs to be decay corrected. (if the isotope verifying is located in this spreadsheet, it needs to be decay corrected). Note, new activity on the prep sheet.
10.10.4 Open new spreadsheet verification folder (located in QA_Public\FORMS\RAD) and select master 3 or 6 point verification (depending on how many standards are made).

10.10.5 Enter calculated value from Decay Corrector (as True value) and value from the spectra print outs (activity on the spectra for the Isotope you are verifying).
10.10.6 Make sure units match.
10.10.7 Standard passes if the mean value is within 5% of certified (true) value, the 1.96 sigma value is within 10% of mean value and the standard reverification acceptablity evaluations are all yes.
10.10.8 Sign bottom of prep sheet and calculation page.
10.10.9 Give to prep supervisior.

10.11 Evaluation of Controls for Trends
10.11.1 Identified controls shall be evaluated for trends at a minimum frequency of 1 month, but as often as deemed by analysts. The controls and trending rules are listed below (see appendix for list of trending rules)
10.11.1.1 FWHM – Trending Rules 2, 3 & 6
10.11.1.2 Centroid (by Channel) – Trending Rules 2, 3 & 6
10.11.1.3 Efficiency – Trending Rules 2, 3 & 6

11.0 PROCEDURE

11.1 For sample preparation reference the applicable preparation SOP.

11.2 Initial Setup
11.2.1 Establish the normal instrument settings for all controls.
11.2.1.1 Detector specific high voltage settings and required polarity are listed in the method software settings.
11.2.2 Pulser quality controls shall be checked before each use of the instrument.

11.3 Counting Samples
11.3.1 In Radcapture, go to Utilities > Export>Choose 'Alphavision' or 'Alphavision1' depending on which instrument the samples were set up on>, enter batch # in the window that pops up, and click ok to export to Alphavision.
11.3.2 In Alphavision, go to Process, select Batch to open the Batch Wizard.
11.3.3 Choose Load from LIMS, and pick the batch.
11.3.4 Choose proper analysis by clicking on the correct isotope.
11.3.5 Select Next.
11.3.6 Click on blank, and then pick blank type (Uu blank, Pu Blank, etc…).
11.3.7 Click on LCS, and then pick LCS type with correct spike number. For amount, use the spike aliquot amount (0.1, 0.2 mL, 0.1326 g, etc) which is printed on the batch paper work.
11.3.8 Select Next.
11.3.9 Live time is count time. Enter correct count time for the batch, select next.
11.3.10 Select Nuclide Library, choose correct ROI and the specified tracer that is printed on the batch paper work.
11.3.11 Select next.
11.3.12 Select correct activity units (DPM, pCi, etc), select Activity concentration,
.
11.3.12.1 Always select TPU Sigma 1, add 5% systematic uncertainty and check the negative activity box. .
11.3.13 Select Next, two times.
11.3.14 Select Print on Completion.
11.3.15 Select Finish.
11.3.16 Click and drag correct detectors to the correct sample ID and select Start Now.

11.3.17 The spectrum will be processed by the software.
11.3.18 For DOE:
11.3.18.1 FWHM of each tracer peak shall be ≤ 100 keV
11.3.18.2 Tracer peak energy for each sample shall be within ± 50keV of the expected energy.
11.3.19 Backgrounds are checked after high activity samples r see section 11.4..

11.4 Samples with a count rate of greater than 1 CPS should be removed from the alpha counting system to prevent contamination of detector(s).
11.4.1 Alpha detectors exposed to samples with count rates greater than 1 CPS should be tagged out-of-service until an empty chamber check can be performed. To perform an empty chamber check, place a clean stainless steel disc in the chamber, establish vacuum, turn on bias and start acquisition. The duration of the background check measurement shall be of sufficient duration (i.e. at least as long as the sample count time) to quantify contamination that may impact routine sample measurements. Note this in the instrument and maintenance log.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery, RPD, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the TestAmerica St. Louis QAM.

12.2 Isotope ROI and libraries are derived from the PCNudat master nuclide library:.

12.2.1 http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

12.3 Any manual integration of a peak or group of peaks must be documented. In all instances where the data system report has been edited or where manual integration has been performed, the operator must clearly identify such edits or manual procedures. Reference SOP ST-QA-0040 for details.
12.4 Blank Population correction may be performed through RadCapture. An interference correction factor from a population of blank samples (min. 20) is entered into the RadCapture database for each isotope/ROI. Corrections should be applied to analyses with known interferences (i.e. thorium-230 interference from thorium-229 tracer) or by client request. When blank pop correction is desired, the analyst will check the "Blank Pop Batch" box during data import into RadCapture before generating an analysis report and uploading to TALs.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) module. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank
13.2.1 Acceptance Criteria:
13.2.1.1 No target analytes may be present in the method blank above the requested limit.
13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements memorandum (CRM).
13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the MB concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than five times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD/RER).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – The batch is re-prepped and re-analyzed for the affected isotope.

13.3.2.3 RPD/RER Duplicate excursion (Batch Precision) – For the RPD/RER, one or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narrated in an NCM. If the sample(s) associated to the batch have activity below the RL, sample(s) may be reported with an NCM,

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD/RER).

13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. Investigate cause (i.e. high target isotopes) and NCM accordingly.

13.5 Sample Result Evaluation

13.5.1 Tracer recovery must be within specified limits. Tracer limits are 30% - 110% unless other wise specified by the client

13.5.2 Tracer/Carrier recovery (low) – Re-extract using a reduced volume or recount for maximum count time to achieve 400 tracer counts

13.5.2.1 Note: QSAS allows for reporting results as quantitative when tracer recoveries are below 30% if:

13.5.2.1.1 the relative uncertainty associated with the tracer recovery is less than 10% (2 sigma)

13.5.2.1.2 spectral resolution requirements are met and there are no indications of spectral interferences (resolution of <100 keV).

13.5.2.1.3 detection limit requirements have been met

13.5.3 Tracer/Carrier recovery (high) – If the Method Blank and LCS are **within limits**, contact a supervisor or manager for further instruction. If the Method Blank and/or LCS has **high recovery**, a reprep is required unless approval is given by the client.

13.5.3.1 Truncation to 100%: Truncation can be done at the clients discretion, or with approval from manager or technical director or based on sample history.

13.5.3.2 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.3.2.1 minimum number of tracer counts

13.5.3.2.2 level of uncertainty

13.5.3.2.3 client project requirements/approval

13.5.4 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.5.5 The following occurances require a dilution to be performed:

13.5.5.1 Dilution level is determined by taking the highest gross counts divided by the count time divided by a factor of 2. (ie: 7200/180/2=1:20)

$$DL = GCts_{High} / t_{count} / 2$$

$$DL = Dilution\ Level$$

$$GCts_{High} = Highest\ Gross\ Counts$$

$$t_{count} = Count\ Time$$

13.5.5.1.1 Tailing – A peak is significantly tailing out side its region of interest (ROI)

13.5.5.1.2 The tracer recovery is low due the high activity of the sample

13.5.5.1.3 Peak Interference – A Peak is observed which is identified as an interference

13.6 Insufficient Sample

13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis an NCM is written and a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Requested Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in ST-QAM.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Measurement Laboratory (EML) HASL 300 28th Edition method A-01-R, Alpha Radioassay

17.2 AlphaVision-32, Alpha Particle Spectrum Acquistion and Analysis for Microsoft Windows and NT, Software Version 5.0 Installation, User Interface and Reference Guide, Ortec (latest version)

17.3 OCTETE Plus, Integrated Alpha-Spectroscopy System Hardware Operating Manual, 777720, Ortec (latest version)

17.4 MAESTRO-32, MCA Emulator for Microsoft Windows, A65-B32 Software User's Manual, 777800, Ortec (latest version)

17.5 U.S. Nuclear Regulatory Commission, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment, Regulatory Guide 4.15.

17.6 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

17.7 TestAmerica, St. Louis Quality Assurance Manual, current revision

17.8 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.9 Decay Radiation Datebase, Version of 5/8/2013; http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

17.10 Associated SOPs, current revisions

17.10.1 ST-PM-0002, Chain of Custody
17.10.2 ST-QA-0002, Standard and Reagent Preparation
17.10.3 ST-QA-0014, Evaluation of Positive and Negative Controls
17.10.4 ST-QA-0036 Non-Conformance Memorandum (NCM) Procedure
17.10.5 ST-QA-0040, Manual Integration Procedure
17.10.6 ST-RC-0040, Total Alpha Emitting Isotopes of Radium
17.10.7 ST-RC-0238, Isotopic Uranium By Eichrom® Uteva Resin For Various Matrices
17.10.8 ST-RC-0210, Determination Of Polonium-210 By Alpha Spectrometry
17.10.9 ST-RC-0240, Isotopic Americium, Curium, Plutionium, Thorium, And Uranium In Various Matrices By Eichrom® Separation Resin

17.10.10 ST-RC-0241, Americium, Plutonium, Curium, And Uranium In Various Matrices By Eichrom® Uteva And Tru Resins (With Vacuum Box System)
17.10.11 ST-RC-0242, Isotopic Thorium, Plutonium And Uranium In Various Matrices By Eichrom® Separation Resins

18.0 MODIFICATIONS TO REFERENCE METHOD

18.1 Energy calibrations checks are performed monthly. Daily pulsar checks are performed in place of the weekly energy calibration checks.
18.2 Backgrounds are determined monthly rather weekly
18.3 CCV's are determined monthly rather that before and after each measurement.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes- Annual Review
19.2 Rev. 8:
- 19.2.1 Section 10 additions
  - 19.2.1.1 Addition of Instrument setup as §10.1
  - 19.2.1.2 Addition of Calibration Quality Control Check as §10.3
  - 19.2.1.3 Addition of calibration acceptance criteria

19.3 Rev. 9:
- 19.3.1 Section 10.5, addition of limits for pulser centroid and pulser energy

19.4 Rev. 10:
- 19.4.1 Section 10:
  - 19.4.1.1 2nd level review for ICV and CCV added to section 10
  - 19.4.1.2 1200 minute setting for acquisition window for special projects
  - 19.4.1.3 Upper control limits for long backgrounds
  - 19.4.1.4 Detector cleaning
- 19.4.2 Section 12: addition of ROI and library reference
- 19.4.3 Section 13: Occurances that require dilution
- 19.4.4 Addition of Attachement 1

19.5 Rev. 11: (8/21/13)
- 19.5.1 Grammatical corrections through out and removal of referencest to QuantIMS and Clouseau
- 19.5.2 Section 10, updated internal calibrations information in section and the calibration process in the lab software throughout
- 19.5.3 Section 10, added new standards verification procedure
- 19.5.4 Section 13, added corrective actions and equation for dilution level
- 19.5.5 Section 15, updated with new verbiage
- 19.5.6 Attachment 1 revised
- 19.5.7 Section 13,removed "See Clouseau NCM for Corrective Action"and added specfic Corrective Actions to the SOP
- 19.5.8 Section 18, added backgrounds will be done monthly and added CCV's will be done monthly.
- 19.5.9 Section 3, Added "keV" definition
- 19.5.10 Section 6, Added Hardware and Software specifics

19.6 Rev. 12: (4/24/15)
- 19.6.1 Section 10.2.1.1, added the energy vs. channel slope should be less than 15 keV per channel. The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Changed 5 channels to 2.5 channels and 40 keV to 20 keV.
- 19.6.2 Section 10.2.2.1, added a minimum of 3000 net counts must be collected for each peak in the calibration.

19.6.3 Section 10.2.3.1, added a minimum of 2000 net counts must be collected for each peak in the ICV. Added the Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibraation peak in the initial calibration (IC).
19.6.4 Section 10.3.1.1, added The Full Width Half Maximum (FWHM) must be less than or equal to 100 keV for each peak. Each peak must fall within 20 keV of the corresponding calibraation peak in the initial calibration (IC).
19.6.5 Added section 10.5
19.6.6 Updated calibration identification in procedure

19.7 Rev. 13: (05/23/2016)
19.7.1 Section 10, removed red detector references
19.7.2 Removed Red Detectors from attachment
19.7.3 Trending evaluation added to section 10
19.7.4 Appendix with trending rules added

19.8 Revision 14 (5/2/17)
19.8.1 Updated hyperlinks throughout SOP.
19.8.2 Replaved all "Red" detectors with "Grren. Red detectors are no longer used.
19.8.3 Changed Trending Rules used from 2,3,and 5 to 2,3,and 6.

19.9 Revsion 15 (3/9/2018)
19.9.1 Grammar and referenced sections fixed throughout
19.9.2 Updated file locations throughout
19.9.3 Section 10.6.3.1- removed part about report generation, this is now done by LIMs
19.9.4 Section 10.7.2- fixed the formatting from previous revision- numbering was wrong causing referenced sections to be wrong.
19.9.5 10.7.3.18- removed redundancy
19.9.6 Section 10.7.4.18- removed re-categroization; we do not re-categroize monthly
19.9.7 Removed text thoughout referencing Quantims
19.9.8 Section 11.4.1- added/removed text to include reference to QSM 5.1
19.9.9 Section 12.4 added- Blank Population section
19.9.10 Section 13.3.2.3- added criteria
19.9.11 Section 13.5.3- removed reference to native correction, we do not do this for actinides methods.

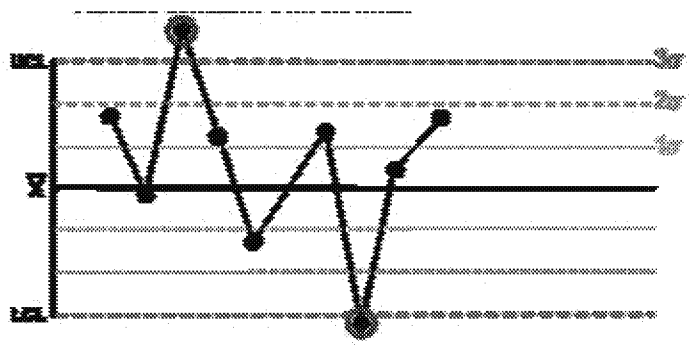
19.10 Revision 16 (9/06/2019) Tech Review: J. Watson/QA Review: M. Ward
19.10.1 Updated cover page with Eurfins header, updated copyright info and CR Manager
19.10.2 Removed requirement for yearly calibrations in Section 10.2

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.

~ Simplest: Single point is considered out of control event

- Corrective action required



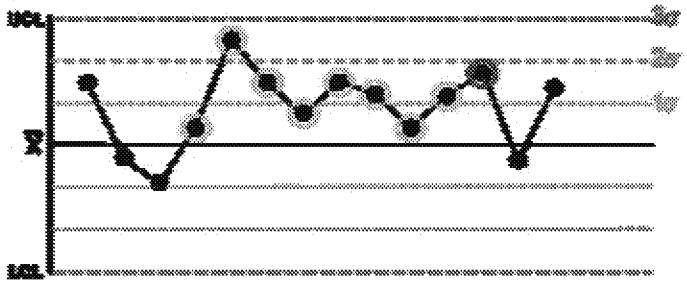
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged <u>bias</u> exists.

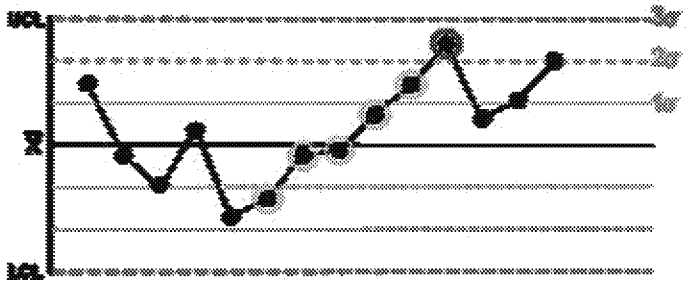
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A <u>trend</u> exists.

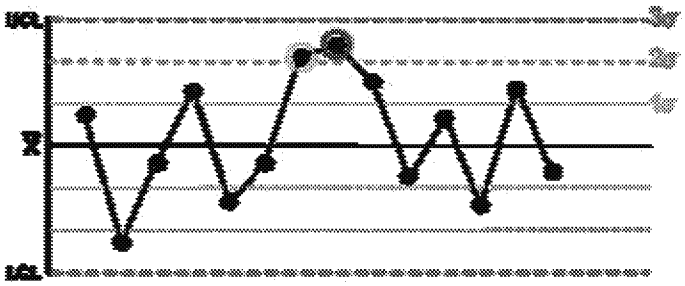
~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

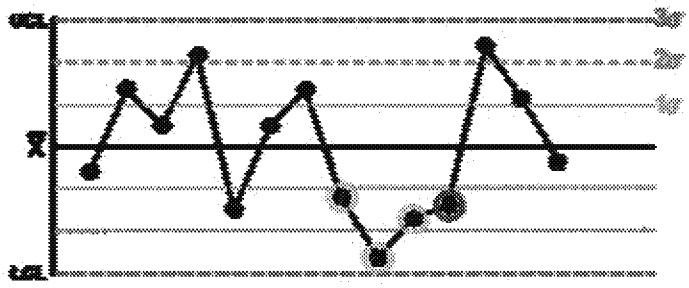
~ Investigate for preventive. May not be CAR



Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

~ No CAR, Statistical trend



Attachment 1

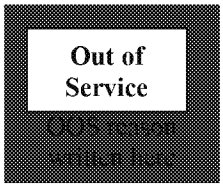
AlphaVision Detector Key

Green – Low Level 0-20 counts

Yellow – Intermediate Low Level 0 – 4 counts
Special Projects and QC samples (Method Blanks and Lab Control Samples) Only

Blue – Ultra Low Level (for Pu/Am/Np)
Special Projects Only

Blue Dot detectors – No Thorium (Am/Cm or Pu)



OOS Tag – Out of Service (OOS)

Title: LIQUID SCINTILLATION COUNTER ANALYSIS

Approvals (Signature/Date):

9-4-19 Jodiy Watson — Date Radiochemistry Count Room Manager	9/5/19 Michael Ridenhower — Date Health & Safety Manager / Coordinator
9/5/19 Kristen Ely — Date Quality Assurance Manager	9/5/19 Jodie Carnes — Date Laboratory Director Rad ops manager

This SOP was previously identified as SOP No. ST-RD-0302 Rev. 18

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Facility Distribution No.: 0 **Distributed To:** See Electronic Distribution Sheet

1.0 SCOPE AND APPLICATION

1.1 This procedure provides instruction for the operation of the Packard Tri-Carb series 3170, and Packard Tri-Carb series 3180 CA/LL Liquid Scintillation Analyzer.

1.2 This procedure applies to all samples that are analyzed using the liquid scintillation counter, namely:

- Carbon-14
- Gross Alpha/Beta
- Iodine-129
- Iron-55
- Lead-210
- Low Energy Beta
- Nickel-59 and 63
- Plutonium-241
- Technetium-99
- Tritium
- Total Activity
- Radon-222
- Selenium-79

1.3 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS)..

2.0 SUMMARY OF METHOD

2.1 The liquid scintillation counter is calibrated with standards containing a known quantity of H^3, C-14, Fe-55, I-129, Ni-59/63, Pb-210, Pu-241, Ra-226, Sr-90, Tc-99, and/or Th-230. Not every instrument is calibrated for every nuclide. However, any instrument used to analyze a given method is calibrated for the nuclide requested.

2.2 Prepared samples are loaded into the sample tray and counted.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.

3.2 Quenching - The interference with the conversion of decay energy to electronic signal in the photo multiplier tubes resulting in a reduction in counting efficiency. This can be caused by materials present in the scintillation solution that interfere with the process leading to the production of light as well as materials present that absorb the light before it reaches the photo multiplier tubes.

4.0 INTERFERENCES

4.1 See individual sample preparation SOPs.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the

method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6.0 EQUIPMENT AND SUPPLIES

6.1 Packard Tri-Carb Series 3170, Packard 3180, Packard Tri-Carb 2550 CA/LL Liquid Scintillation Analyzers.

6.2 Liquid Scintillation Counter vials

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water

7.3 Unquenched standards:
- 7.3.1 Background Standard
- 7.3.2 Unquenched C -14 standard (this standard is also used for Se-79)
- 7.3.3 Unquenched I-131 standard
- 7.3.4 Unquenched Fe-55 standard
- 7.3.5 Unquenched I-129 standard
- 7.3.6 Unquenched Ni-59/63 standard
- 7.3.7 Unquenched Pb-210 standard
- 7.3.8 Unquenched Pu-241 standard
- 7.3.9 Unquenched Tc-99 standard
- 7.3.10 Unquenched Tc-99 for Total Activity standard
- 7.3.11 Unquenched H3 standard
- 7.3.12 Unquenched Pm-147 standard
- 7.3.13 Unquenched Sr-90 standard
- 7.3.14 Unquenched Th-230 standard
- 7.3.15 Unquenched Ra-226 standard

7.4 For Quench curves:
- 7.4.1 Quenched C-14 standard (this standard is also used for Se-79)
- 7.4.2 Quenched I-131 standard
- 7.4.3 Quenched Fe-55 standard
- 7.4.4 Quenched I-129 standard
- 7.4.5 Quenched Ni-59/63 standard
- 7.4.6 Quenched Pb-210 standard
- 7.4.7 Quenched Pu-241 standard

7.4.8 Quenched Tc-99 standard
7.4.9 Quenched Tc-99 for Total Activity standard
7.4.10 Quenched H3 standard
7.4.11 Quenched Pm-147 standard
7.4.12 Quenched Sr-90 standard
7.4.13 Quenched Th-230 standard

7.5 Standards may contain varying activities depending on the supplier and concentration availability of the nuclide of interest. However, all standards in a quench curve are counted until a minimum of 10,000 counts are collected.

7.6 Sample to cocktail ratios: See Attachment 1 of this SOP.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.

8.2 See individual sample preparation SOP for specific sample handling and storage requirements.

9.0 QUALITY CONTROL

9.1 See individual sample preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), Matrix Spike (MS) and a sample duplicate (DU) unless otherwise noted in specific sample preparation SOPs (i.e. Rn-222, ST-RC-0222). In the event that there is insufficient sample to analyze a sample duplicate, a LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.3.2 A method blank (DI water) must be prepared with every sample batch.

9.4 **Laboratory Control Sample**

9.4.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 A LCS must be prepared with every sample batch.

9.5 **Matrix Spike**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Initial Calibration:

10.1.1 Prepare a quench curve for the nuclide of interest.

10.1.1.1 The process for quench curve preparation can be found on the print out of the previous calibration file (\\tafs\lab\st louis\public\rad\upload)..

10.1.2 Frequency:

10.1.2.1 Calibration/Quench curves shall be performed prior to initial use, following repair or replacement of a key detector component when subsequent performance checks indicate a change in performance, after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in the instrument response, or when indicated by corrective actions.

10.1.3 Acceptance Criteria:

10.1.3.1 A minimum of 10,000 counts must be obtained for each data point

10.1.3.2 For quench curves, the coefficient of determination (r^2) must be greater than 0.99 (corresponds to correlation coefficient, r, > 0.995) or each data point must be less than 7.5% relative to the calibrated value calculated from the fitted curve (residual). For crosstalk (spillover) curves, there is no specific requirement except the associated quench curve must meet the requirement above.

10.1.3.2.1 Except in specific instances, it is NOT acceptable to remove points from a calibration curve in order to meet criteria. There must be some other demonstrable reason to remove a point. Consult with your Supervisor or Technical Director for guidance.

10.1.3.3 For fixed efficiency calibrations, an average of three points shall have a relative standard deviation (RSD) of ≤ 10%. (i.e. Rn-222).

10.2 Initial Calibration Verification (ICV)

10.2.1 The initial calibration verification standard is a different standard source than the one used for the initial calibration.

10.2.2 Validate the quench curve by counting at least three second source standards.

10.2.2.1 The process of preparing validation standards is found on the print out of the previous calibration file (\\tafs\lab\st louis\public\rad\upload). The verification standards should contain an unquenched standard as well as quenched standards that represent the quench range of live samples.

10.2.3 Frequency:

10.2.3.1 An ICV is performed with every initial calibration.

10.2.4 Acceptance Criteria:

10.2.4.1 Mean Recovery +/- 10% of the known value.

10.2.4.1.1 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial

calibration curve or ICV standard.

10.2.4.1.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.3 Continuing Calibration:

10.3.1 Load standards and use the appropriate protocol flag as listed on the status page "Daily QC". The instrument will perform the required tests and the results are printed and saved to the Quality Control Table.

10.3.2 Frequency:

10.3.2.1 Quality control checks are performed for each day of use for the nuclides which measurements are being performed.

10.3.3 Acceptance Criteria:

10.3.3.1 The instruments are set at 2 sigma for a warning and 3 sigma for out of control limits.

10.3.3.2 If a Continuing Check fails and the analyst can document the reason for failure (e.g. carry-over from the previous sample, etc.) then a second check may be analyzed without any adjustments to the instrument. If this check meets criteria then sample analysis may continue.

10.3.3.3 If this second check does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require calibration.

10.4 Evaluation of Controls for Trends

10.4.1 Identified controls shall be evaluated for trends at a minimum frequency of one month, but as often as determined by the analyst (see Appendix for list of trending rules)

10.4.2 Background vial counts – trending rules 2,3 & 6

10.4.3 C-14 Vial Counts – trending rules 2,3 & 6

10.4.4 H-3 Vial Counts – trending rules 2,3 & 6

11.0 PROCEDURE

11.1 Confirm the following:

11.1.1 Computer software is loaded and operational.

11.1.2 Printer is on-line.

11.2 Load sample vials by lifting the instrument cover and placing samples in tray with flag facing towards the left.

11.2.1 While placing the samples in the instrument, inspect the samples to ensure proper loading (mixing) of cocktail in the vial. If proper mixing is not observed (e.g. separation of phases is seen or in the case of InstaGel cocktail the sample is not gelled), corrective action should be taken. Such corrective action may include shaking the vial to promote proper loading and then recounting after additional dark-adaption. If unsure of appropriate corrective action, consult your supervisor or the Technical Director..

11.3 Samples are counted from left to right. QC checks are run daily. Prior to starting samples, make sure the daily QC has run and is acceptable.

11.4 Position 1 in the sample tray is reserved for the background vial.

11.5 Log the following information into the Run Log:
- 11.5.1 Date
- 11.5.2 Batch number
- 11.5.3 Sample I.D.
- 11.5.4 Protocol No.
- 11.5.5 Count length
- 11.5.6 Type of analysis
- 11.5.7 Operator's initials

11.6 Close instrument cover and allow sample vials to dark-adapt per the cocktail manufacturer's instructions, typically 30 minutes.

11.7 For the Packard 3170 LSC and 3180 LSC:
- 11.7.1 Maximize the instrument window. Double click on the flag icon which has the protocol number that needs to be run. Click on the count conditions tab at the top of the analysis window. Enter the correct count time in the count time field.
- 11.7.2 Click on the Worklist tab at the top of the analysis window. Scan the samples from the Rad Capture paperwork.
- 11.7.3 Verify sample tray is in the correct order, starting with the background, blank, LCS, followed by the samples. Check tray order with the order appearing on the screen version of the worklist.
- 11.7.4 Click on the green start flag in the upper left corner of the instrument window.
- 11.7.5 Upon completion of counting, results will be sent to printer. The data is sent to the appropriate instrument folder.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations are performed in RadCapture (TestAmerica St. Louis custom software). These calculations are printed directly from the software and are included the TestAmerica St. Louis ST-QAM.

12.2 Commonly used calculations (e.g. % recovery, RPD, RER, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank
- 13.2.1 Acceptance Criteria:
  - 13.2.1.1 No target analytes may be present in the method blank above the requested limit.
  - 13.2.1.2 Project specific requirements, if more stringent than our routine procedure (e.g. no target analytes present above ½ RL), will be noted on the client requirements memorandum (CRM) or in the project notes in LIMs.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
  - 13.2.2.1 <u>Method Blank Contamination</u> – Blank contamination above the RL (> ½ RL for some programs – see specific CRM or project notes in LIMs for details) requires re-prep of batch unless all associated samples are < RL or greater than five times the amount detected in the method blank. An NCM is written to

document the excursion.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD/RER).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples that are non-detect (activity below the RL) may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the analyte recovered high in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – batch is re-prepped and re-analyzed.

13.4 RPD/RER Duplicate excursion

13.4.1 For the RPD/RER, one or both must be within acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a re-prep of the samples. If the sample(s) have a physical matrix issue (i.e. non-homogenous), results can be reported with an NCM. If the sample(s) fail RPD/RER criteria after a re-prep and no matrix issue is observed, sample(s) may be reported with client approval and narrated in an NCM. If the sample(s) associated to the batch have activity below the RL, sample(s) may be reported with an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 Analyte(s) should be within control limits for accuracy (%Recovery) and precision (RPD/RER).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Recovery excursion may not necessarily warrant corrective action other than narration. If affected analyte activity in the original sample is greater than five times the amount spiked, percent recovery information is ineffective. Data is reported with an NCM. If the excursion is due to physically evident matrix interferences, the data is reported with an NCM (the physical interferences must be described in the NCM). If there is no evidence of interference and the RPD/RER as well as spike recoveries are out outside limits, sample re-prep and re-analysis are required.

13.6 Sample result evaluation

13.6.1 Tracer recovery must be within specified limits.
Tracer/Carrier recovery low– batch is re-prepped and re-analyzed.

13.6.2 Tracer/Carrier recovery high–batch is re-prepped and re-analyzed. Samples that are non-detect may be reported with an NCM.

13.6.2.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.6.2.1.1 Minimum number of tracer counts

13.6.2.1.2 Level of uncertainty

13.6.2.1.3 Client project requirements/approval

13.6.2.1.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Iodine	Nickel	Lead	Iron
101%	97%	110%	101%

13.6.3 Review the quench parameter to ensure it is within the calibration range.

13.6.3.1 If it is outside the upper end of the range, a small amount (e.g. 10 uL) of nitromethane may be added to the sample and the sample recounted (after waiting again for dark adaption).

13.6.3.2 If the quench parameter is outside the lower end of the range, the sample should be re-prepared and reanalyzed, possibly using a smaller sample aliquot to reduce the interference. There are instances where use of a quench value outside the calibration range may be acceptable with Manager or Technical Director approval and appropriate NCM.

13.6.4 Evaluate spectra for interferences, peak shape, etc. against a known source (either the LCS or the instrument reference manual).

13.6.5 These expectations will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.7 Insufficient Sample

13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report case narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."

16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator."

17.0 REFERENCES

17.1 EPA-600/4-80-032. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Environmental Monitoring and support Laboratory, Section 10, Method 906, "Tritium in Drinking Water", August, 1980

17.2 ANSI N42.15 – American National Standard Check Sources for verification of Liquid Scintillation Counting Systems.

17.3 Standard Methods for the Examination of Water and Wastewater. 19th Edition, Method 7500-1B Mod "Precipitation Method", 1985

17.4 EPA 520/5-84-006, EERF Radiochemistry Procedures Manual, Method C-01-1, "Radiochemical Determination of Carbon-14 in Aqueous Samples", August 1984

17.5 Eichrom Technologies, Inc. Analytical Procedures, TCS01, "TC-99 in Soil", April, 2002

17.6 Eichrom Technologies, Inc. Analytical Procedures,TCW01, TC-99 in Water, April 2002

17.7 Eichrom Technologies, Inc. Analytical Procedures,OTW01, Pb-210 in Water, April 2001

17.8 Eichrom Technologies, Inc. Analytical Procedures,OTS01, Pb-210 in Soil, August, 2002

17.9 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.10 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.11 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

17.12 Associated SOPs, current revisions;

17.12.1 ST-PM-0002, Sample Receipt and Custody
17.12.2 ST-QA-0002, Standard and Reagent Preparation
17.12.3 ST-QA-0014, Evaluation of Positive and Negative Controls
17.12.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.12.5 ST-RC-0030, The Determination of Tritium in Water (and Other Fluids), Soil and Silica
17.12.6 ST-RC-0042, SM 7500 I-B Iodine-129 in Water
17.12.7 ST-RC-0055, Determination of Iron-55, Nickel-59 and Nickel-63 by Liquid Scintillation Spectrometry

17.12.8 ST-RC-0125, Determination of Technetium-99 using Eichrom® TEVA Resin
17.12.9 ST-RC-0211, Determination of Lead-210 by Liquid Scintillation Counting
17.12.10 ST-RC-0245 , The Determination of Pu-241 by Liquid Scintillation Counting
17.12.11 ST-RC-0247, Promethium-147 and Samarium-151 analysis Utilizing Lanthide Resin Separation Procedure
17.12.12 ST-RC-0222, "Determination of Radon-222-Adapted from EPA 913.0 & SM7500-Rn"
17.11.14 ST-RC-0302, Gross Alpha/Beta and Low Energy Beta by Liquid Scintillation Spectrometry.
17.11.15 ST-RC-0079, Radiochemistry Determination of Selenium-79

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA method 906 is a drinking water method, and as such formulated its' Tritium calculation to reflect counts per minute (CPM) rather than disintegrations per minute (dpm). In true drinking water samples, it is acceptable to use CPM since drinking water samples should have little effect on efficiencies. However, EPA method 906 is commonly referenced for wastewater samples, and even silica gels and soil matrices. For these matrices, there can be a significant difference in efficiency and thus using dpm provides a more accurate result.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Added Promethium back to the list of standards in section 7.3.11 and 7.3.10.
19.2 Rev 13:
- 19.2.1 Added Iodine-131 and Total Activity to section 1.2.
- 19.2.2 Added Iodine-131 to section 2.1.
- 19.2.3 Added section 7.6 and attachment 1 to document sample to cocktail ratios
- 19.2.4 Removed redundant section 18.2

19.3 Rev 14:
- 19.3.1 Replaced TALS and Clouseau references with LIMS
- 19.3.2 Removed Packard 2550 instructions from section 11
- 19.3.3 Added native carrier to section 13
- 19.3.4 Removed Clouseau NCM in Section 13 and added specific corrective actions.

19.4 Rev15: (08/26/2015)
- 19.4.1 Moved spectral review to section 13 (from section 11).
- 19.4.2 Added quench parameter review to section 13.
- 19.4.3 Added reference to Gross Alpha/Beta and Low Energy Beta along with associated standards (Sr-90, Th-230).
- 19.4.4 Added calibration evaluation information regarding crosstalk curves to section 10.1.3. Also, clarified correlation coefficient (r) vs coefficient of determination (r^2).
- 19.4.5 Corrected 9.1 to "individual sample preparation SOPs" from "actinides SOPs".
- 19.4.6 Added reference to new SOP ST-RC-0302

19.5 Annual Review – No Changes (01/26/2016)
19.6 Rev 16: (02/07/2017)
- 19.6.1 Added ANSI reference to Section 17 (Corrected reference 5/2/17-mw)
- 19.6.2 Added Trending Rules to Section 10.4 and Attachment 1
- 19.6.3 Made former attachment 1, Attachment 2
- 19.6.4 Changed rule 5 to rule 6 in section 10.4

19.7 Rev 17: (10/16/2017)
- 19.7.1 Section 7: removed reference to Milli Q system for DI
- 19.7.2 Section 9: changed acronym for sample duplicate, from "SD" to "DU", to match LIMS
- 19.7.3 Section 11: added the inspection of samples to ensure proper mixing of cocktail in vial.

19.8 Revision 18 (4/20/2018) Tech Review: R. Mueller/QA Review: M Ward
- 19.8.1 Section 1.1- removed the 2550 instrument

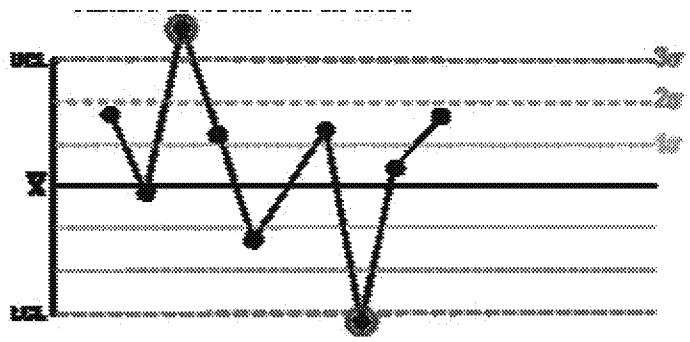
19.8.2 Section 1.2- added Rn-222 and Selenium-79
19.8.3 Updated Section 7 to incude Rn-222 and Se-79
19.8.4 Section 9.2.3- referenced Rn-222 SOP due to different QC requirements
19.8.5 Fixed grammar and file locations throughout
19.8.6 Section 10.1.3.2.1- made a sub-point
19.8.7 Section 10.1.3.3 added- Fixed efficiency calibrations (i.e. Rn-222)
19.8.8 Section 13.2- added Method Blank criteria
19.8.9 Added Section 13.4 – RPD/RER criteria
19.8.10 Section 13.6.2.1.4 – Updated statistical limits for native correction from the last year
19.8.11 Section 17 updated to include Radon SOP ST-RC-0222 and Selenium-79 SOP – ST-RC-0079

19.9 Revision 19 (9/06/2019) Tech Review: Jody Watson/QA Review: M. Ward
19.9.1 .Updated cover page: added Eurfins logo, updated Copyright info; Updated CR Manager
19.9.2 Section 10.1 removed annual calibration requirement

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.
~ Simplest: Single point is considered out of control event

- Corrective action required



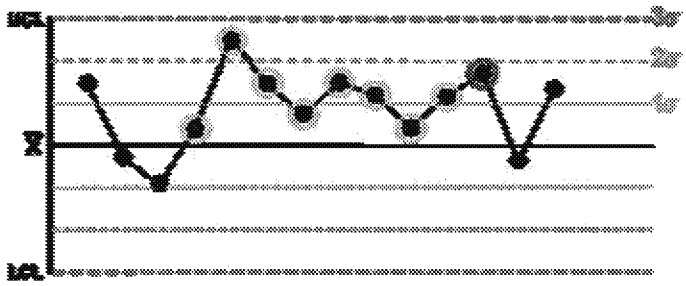
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged bias exists.

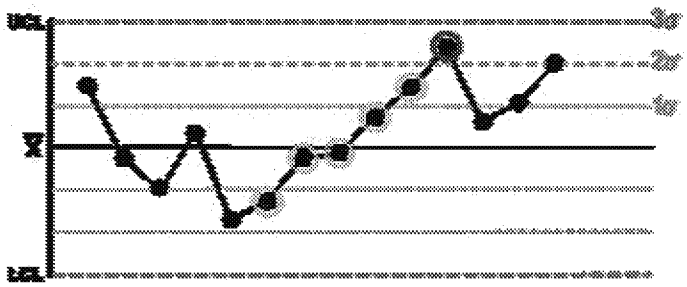
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A trend exists.

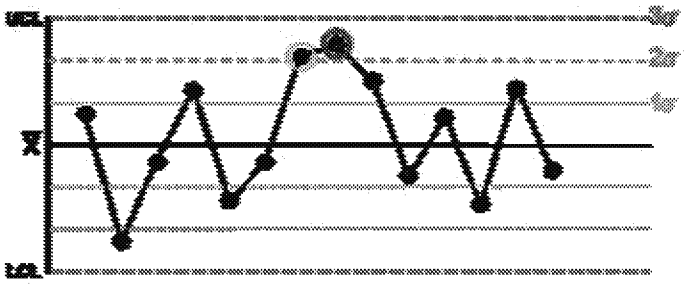
~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

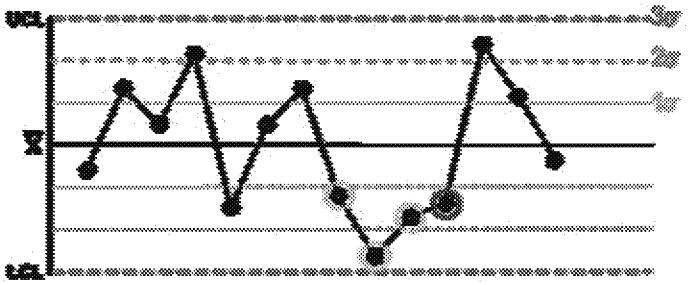
  ~ Investigate for preventive. May not be CAR



Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

  ~ No CAR, Statistical trend



Attachment 2

LIQUID SCINT. SAMPLE TO COCKTAIL RATIOS

ST-RC-0015 | Total Activity

Solid and Semi-solid Samples

0.25 - 3 mL	Sample
15 - 17 mL	Cocktail
18 mL	Final

Aqueous Samples

0.25 - 3 mL	Sample
15 - 17 mL	Cocktail
18 mL	Final

Oil Samples

3 mL	Sample
15 mL	Cocktail
18 mL	Final

ST-RC-0030 | Tritium

Steam Distillation (Water); Silica Gel; Solids (soil, filters/swipes); Direct Method - Water

10 mL	Sample
10 mL	Cocktail
20 mL	Final

Direct Method - Oil

1 - 2 mL	Sample
18 - 19 mL	Cocktail
20 mL	Final

ST-RC-0031 | Tritium Cryo

10 mL	Sample must dilute to 10 mL total if 10 mL of sample not present
10 mL	Cocktail
20 mL	Final

ST-RC-0042 | I-129

Carrier Determination by ICP-MS

4.9 mL	Sample
15 mL	Cocktail
19.9 mL	Final

gravimetric carrier

5 mL	Sample
15 mL	Cocktail
20 mL	Final

ST-RC-0055 | I-55 & Ni-59/63

Iron

4.9 mL	Sample
15 mL	Cocktail
19.9 mL	Final

Nickel

3.9 mL	Sample
16 mL	Cocktail
19.9 mL	Final

ST-RD-0302_LqScint-Sample2CocktailRatio.xls Rev (0)

LIQUID SCINT. SAMPLE TO COCKTAIL RATIOS

ST-RC-0050	C-14
10 mL	Sample
10 mL	Cocktail
20 mL	Final

ST-RC-0125	Tc-99
2 mL	TEVA resin
5 mL	dI Water
10 mL	Cocktail
17 mL	Final

ST-RC-0211	Pb-210
5 mL	Sample
15 mL	Cocktail
20 mL	Final

ST-RC-0215	Pu-241
4 mL	Sample 0.1 mL dI water rinse used
16 mL	Cocktail
20 mL	Final

ST-RC-0247	Pm-147
10 mL	Sample
10 mL	Cocktail
20 mL	Final

ST-RD-0302_LqScint-Sample2CocktailRatio.xls Rev (0)

Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

Approvals (Signature/Date):

11-5-19 Jody Watson Date Radiochemistry Count Room Manager	11/6/19 Michael Ridenhower Date Health & Safety Manager / Coordinator
11/6/19 Kristen Ely Date Quality Assurance Manager	11/6/19 Jodie Carnes Date Laboratory Manager

This SOP was previously identified as SOP No. ST-RD-0403 Rev. 19

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs radium-226/228, strontium-89/90, gross alpha/beta, and chlorine 36.

1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; Standard Method 7110C and DOE EML HASL 300 method, Ba-01-R, Sr-02 and Sr-03-RC.

1.3 The SOP applies to GFPC analysis of liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.

3.2 IQC - a computerized Quality Control Program where the counting results of Daily Radioactive check sources and Daily Background checks are entered and compared to statistical average data. A measurement within ± 3 standard deviations indicates the detector is operating within acceptable parameters.

3.3 αLL - discriminator setting indicating the alpha lower voltage limit.

3.4 Alpha Voltage Only - detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.

3.5 αUL - discriminator setting indicating the instruments alpha upper voltage limit.

3.6 βLL - discriminator setting indicating the beta lower voltage limit.

3.7 βUL - discriminator setting indicating the beta upper voltage limit.

3.8 Crosstalk - a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.

3.9 Plateau - a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.

3.10 LB4100 – LBPC (Low background Gas Flow Proportional Counting instrument).

4.0 INTERFERENCES

4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.

4.1.1 If a sample reaches 10,000 counts, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits.

4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm2. Therefore, the maximum acceptable mass density is typically 5 mg/cm2 or less that 100 mg for a 2" planchet.

4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm2 or greater.

4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1.1 None.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Silver Nitrate	Poison Corrosive Oxidizer	$0.01^{g}/_{m3}$ (TWA) for silver, metal dust, and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact may cause redness, pain, and severe burning. Eye contact can cause blurred vision, redness, and pain.
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms may include irritation to the respiratory tract. Ingestion symptoms may include pain in the mouth, chest, and abdomen with coughing, vomiting, and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.

6.2 Gas mixture, 90% argon, 10% Methane

6.3 Blank planchets

6.4 PC based data acquisition system, IQC software

6.5 Centrifuge tubes
6.6 Centrifuge
6.7 Vortex
6.8 Pipettes, Eppendorf or equivalent
6.9 Pipette, disposable

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision

7.2 Radioactive sources to measure beta radiation,: Sr-90

7.3 Radioactive sources to measure alpha radiation: Am-241, Am-243, Th-230 and Ra-226

7.4 Deionized Water (DI).

7.5 Silver nitrate ($AgNO_3$), 0.5 N

7.6 Sodium chloride (NaCl), crystals

7.7 Sodium chloride (NaCl), 0.5 N

7.7.1 Add 50 mL of DI water to a 100 mL volumetric, add 5.84 g of NaCl, dilute to 100 mL, cap and shake to dissolve. Adjust volume to 100 mL with DI water.

7.8 Ammonium hydroxide (NH_4OH), concentrated, 28 N

7.9 Ammonium hydroxide (NH_4OH), 5 %

7.9.1 Add 25 mL of concentrated Ammonium Hydroxide to 475 mL of DI water. **CAUTION – Ammonium hydroxide is corrosive. Mist and vapor cause burns to every area of contact.**

7.10 Cl-36: At least four sodium chloride standards are prepared for calibration.
7.10.1 Add 10 mL of DI water to 4 centrifuge tubes.
7.10.2 Add 0.500 mL of 0.5 N sodium chloride carrier solutions to each centrifuge tube. Swirl to mix.
7.10.3 Add 2 drops of 5 % ammonium hydroxide solution, swirl to mix.
7.10.4 Add 12 mL of 0.5 N silver nitrate solution to each centrifuge tube.
7.10.5 Vortex for 30 seconds.
7.10.6 Centrifuge and decant supernate to waste.
7.10.7 Proceed to section 11.4, Planchet Preparation of Silver Chloride Precipitation of SOP ST-RC-0036.
7.10.8 Average the four weights for the sodium chloride carrier solution, record the standardized weight in the log book and on the bottle.
7.10.9 NOTE: It may be necessary to use more than 0.500 mL of carrier in some large water samples or calibrate a 4 N sodium chloride carrier solution. The efficiency of the detectors will have to be calculated using the heavier sodium chloride carrier solution.

7.10.10 Prepare four sodium chloride calibration samples as in Section 7.10 but add a known amount of Cl-36 to each tube before the sodium chloride carrier is added. Analyze samples by GFPC and determine detector efficiency as per Section 12, Data Analysis and Calculations.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041and ST-RC -0050, for more detailed information.

9.0 QUALITY CONTROL

9.1 See GFPC preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same reagent lots.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS is a blank matrix spiked with a known amount of target analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every sample batch.

9.5 **Matrix Spike**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample, processed simultaneously with, and under the same conditions as, samples through all steps of the analytical process

to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Additional preventative maintenance can be found in ST-QA-0024.

10.2 Voltage Plateau Determination

10.2.1 **Frequency**:

10.2.1.1 Performed as a part of the Intial Calibration.

10.2.2 **Voltage Plateau Determination on Protean MPC 9604**

10.2.2.1 Place the Sr 90 source or sources in the detector drawer.

10.2.2.2 Select detector of interest on the computer screen.

10.2.2.3 Click Plateau under Count Method.

10.2.2.4 Set time to accumilate at least 10,000 counts for distributed Sr90 source.

10.2.2.5 Select A, B. C, D.

10.2.2.6 Click OK.

10.2.2.7 When count is complete select Plateau under instrument Specific.

10.2.2.8 Set Beta appropriate voltage with arrows </>. Evalulate and Print report.

10.2.3 **Criteria for Plateaus for Protean MPC 9604**

10.2.3.1 Acquire 40 data points in 30V increments beginning at 705V and ending at 1875V. Slope should be no more than 5%.

10.3 **Discriminator Settings**

10.3.1 **Frequency:**

10.3.1.1 Performed as a part of the Intial Calibration.

10.3.2 **Discrimator Settings on Protean MPC 9604**

10.3.2.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and/or Po-210 sources

10.3.2.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.

10.4 **Initial Calibration (IC)**:

10.4.1 **Frequency**:

10.4.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated prior to initial use, following repair or replacement of a key detector part when subsequent performance checks indicate a change in performance after modification of system parameters which affect instrument response, when performance checks (e.g. CCV) indicate a change in the instrument response, or when indicated by corrective actions.

10.4.2 The specific calibration source preparations can be found in the file containing the previous calibration.

10.4.3 All nuclide sources shall be NIST traceable.

10.4.4 The efficiency calibration shall consist of at least seven mass attenuated calibration standards, unless a single point source efficiency is to be determined.

10.4.5 Alpha, Beta Ra226 - at least seven mass attenuated calibration standards

10.4.5.1 For gross alpha, use Th-230 in the nitrate form (spiked into varying amounts of salt solution, converted to nitrate) .

10.4.5.2 For Ra-226, use Ra-226 standard in sulfate form (co-precipitated with varying amounts of barium sulfate).

10.4.5.3 For gross beta, Sr-90, Total Beta Strontium, and Ra-228, use Sr-90/Y90 in the nitrate form (spiked into varying amounts of salt solution, converted to nitrate).

10.4.5.4 For gross alpha by co-precipitation, use Th-230 standard (spiked into varying amounts of iron [hydroxide] and barium [sulfate] carrier, dissolved into nitric acid, and dried onto the planchet.

10.4.6 Air Filter – single point calibration

10.4.6.1 For alpha use Am-241

10.4.6.2 For beta use Sr-90

10.4.7 Cl-36 – Averaged 4 point calibration.

10.4.7.1 For Cl-36 use Cl-36

10.4.8 The standards shall have enough activity to generate at least 10,000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5,000 counts per second.

10.4.9.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.

10.4.9 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.

10.4.9.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.

10.4.10 Each standard shall be counted in every detector to be calibrated.

10.4.11 **Alpha to Beta Crosstalk Determination**

10.4.11.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.

10.4.11.2 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.

10.4.11.3 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.

10.4.11.4 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.

10.4.11.4.1.1 In this manner the crosstalk factor can be determined for any given mass.

10.4.11.5 The equation of the curve shall be determined using polynomial functions.

10.4.11.6 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.4.12 **Beta to Alpha Crosstalk Determination**

10.4.12.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.

10.4.12.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.

10.4.12.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

10.4.13 **IC Criteria:**

10.4.13.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.

10.4.13.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.

10.4.13.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.

10.4.13.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.

10.4.13.5 Points that are visual outliers or demonstrate greater than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.

10.4.13.6 The coefficient of determination (r^2) shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.5 **Independent Calibration Verification (ICV)**

10.5.1 **Frequency**:

10.5.1.1 Performed with every intial calibration

10.5.2 GFPC initial calibrations must be verified by a second source standard.

10.5.3 The ICV standard is NIST traceable.

10.5.4 The ICV is counted to accumulate at least 5,000 counts.

10.5.5 **ICV Criteria**:

10.5.5.1 Prepare 3 verification sources varying in expected mass (low, medium and high) within the calibration range of the curve, unless a single point source is to be determined.
10.5.5.2 Alpha and Beta 3 sources
10.5.5.3 Ra226 Minimum of one source
10.5.5.4 Air Filter Minimum of one source
10.5.5.5 Cl-36 – Minimum of one source
10.5.5.6 The source/standard used for the ICV shall be from an independent second source as defined within the laboratory Quality Assurance Manual."
10.5.9.7.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.
10.5.5.7 Count the secondary source in all detectors that were calibrated.
10.5.5.8 Calculate the results in terms of percentage recovery.
10.5.5.9 Calculate the mean results of all masses across each detector.
10.5.5.10 **Criteria:**
10.5.9.11.1 Individual points are within 30 percent of the true value
10.5.9.11.2 The mean result of all masses across all detectors is less than 10 percent.
10.5.9.11.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.6 **Setting Performance Check Criteria After Calibration**
10.6.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.
10.6.2 The limits for the background check sample will be established with five points from four months. Every month the oldest months points will be removed and points from the current month will be added.
10.6.3 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.
10.6.4 The limits for alpha/beta check sources will be a running average of the four months post calibration.
10.6.4.1 The limits are to be documented.
10.6.4.2 The limits will be re-established monthly at the following frequency
10.6.4.2.1.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.
10.6.4.2.1.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.
10.6.4.2.1.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.
10.6.4.2.1.4 4th month – take first five data points from new month and five points each from the previous three months.
10.6.4.3 Limits are set.

10.7 **Long Monthly Background (ICB)**
10.7.1 **Frequency**:
10.7.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)
10.7.1.2 Minimum count time: 1000 minutes.

10.7.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.

10.7.2.1 Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.

10.7.3 Check that instrument settings are as specified in 11.1.

10.7.4 **Orange, Protean, Purple, Red and Blue Long Background Count Set-Up**

10.7.4.1 Select detector 0
10.7.4.2 Select 'sources'→ Start
10.7.4.3 Select 'ICB' by clicking on the Type List arrow.
10.7.4.4 Ensure count time is set to 1000 minutes.
10.7.4.5 Select 'start'
10.7.4.6 Continue these steps with detectors 1-23.
10.7.4.7 Review the data for acceptance when the backgrounds are complete.

10.7.5 **Printing Orange, Protean, Red, Purple and Blue Long Backgrounds**

10.7.5.1 Select 'Data
10.7.5.2 Select 'Source Count Data'
10.7.5.3 Select QC Chart →ICB; Monthly Background
10.7.5.4 Select 'This Range' and enter the date range that the Long Backgrounds (ICBs) were performed and select Refresh
10.7.5.5 Select 'Refresh'
10.7.5.6 Select 'Source Count Summary' under Reports
10.7.5.7 Select source count detail from the Report menu and 'Print'
10.7.5.8 Export to TALs using the Rad Instrument Exporter by editing the file name with _ICB (ex. 10102018_ICB)

10.7.6 **Long Background Criteria:**

10.7.6.1 Long backgrounds are evaluated at ± 3 sigma.
10.7.6.2 The data report is evaluated per detector.
10.7.6.3 If a detector is above this limit, discard planchet.
10.7.6.4 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
10.7.6.5 Place a clean planchet in the holder and repeat steps for that detector (s) only.
10.7.6.6 Perform a new background.
Note: The detector is tagged with an out of service tag noted with ICB and date. Detector is out of service until a successful background has been achieved.

10.8 Evaluation of Controls for Trends

10.8.1 Identified controls shall be evaluated for trends at a minimum frequency of 1 month, but as often as deemed by analysts. The controls and trending rules are listed below (see appendix for list of trending rules)

10.8.1.1 Alpha background counts – Trending Rules 2, 3 & 6
10.8.1.2 Alpha source counts – Trending Rules 2, 3 & 6
10.8.1.3 Beta background counts – Trending Rules 2, 3 & 6
10.8.1.4 Beta source counts – Trending Rules 2, 3 & 6

11.0 PROCEDURES

11.1 Initial Setup

11.1.1 Check the normal instrument settings for all controls as described below:

11.1.1.1 Tank Flow 8 psi

11.1.1.2 Flow Cells >/= 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.

11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Gas Flow Proportional counting system remains as set by the manufacturer and does not require adjustment.

11.1.3 If counting gas has just been turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes in P-10 tank log book.

11.2 Record date of Daily Background and Check Source Data in Maintenance Tracker found in the QA Public Folder..

11.3 Maintenance

11.3.1 Change out the counting gas when the gauge reads under 500 psi. . Record gas tank changes in P-10 tank log book.

11.3.2 Allow gas to purge a minimum of 30 minutes prior to operation.

11.3.3 Background and checksource checks are required following a gas bottle change.

11.4 Data Acquisition: Daily Background Check and Source Check

11.4.1 **Daily Background Check**: **<u>Orange, Protean, Red, Purple and Blue</u> Instruments**

11.4.1.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.

11.4.1.2 Select detector 0.

11.4.1.3 Select 'sources ' → Start

11.4.1.4 Select or scan 'CCB' by clicking on the Type List arrows.

11.4.1.5 Select 'CCB' by clicking on the file list arrows for purple.

11.4.1.6 Select 'start'

11.4.1.7 Repeat these steps with detectors 1-23.

11.4.2 **Daily Background Criteria:**

11.4.2.1 Review the IQC report for each detector.

11.4.2.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-count.

11.4.2.1.2 Tag detector out of service with an OOS tag noted with "Bkg RC".

11.4.2.1.3 If detector fails Background re-count tag detector with an out of service tag noted with date to indicate that the detector is out of service for the day.

11.4.3 **Daily Source Check <u>Orange, Protean, Red, Purple and Blue</u> Instrument**:

11.4.3.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.

11.4.3.2 Select detector.

11.4.3.3 Select 'sources' → Start

11.4.3.4 Scan the corresponding alpha or beta source ID into the Type List. Use the alpha barcode list for starting alpha souces and the beta barcode list for starting beta sources.

11.4.3.5 Select 'start'

11.4.3.6 Repeat these steps for detectors 0-7 for alpha and 8-15 for beta using the correlating detector number and the corresponding source ID..

11.4.3.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 0-7 and place alpha sources in detectors 8-15.

11.4.3.8 . Repeat steps 11.5.1.2 to 11.5.1.6 with beta sources in detectors 0-7 and alpha souces in detectors 8-15.

11.4.3.9 Repeat steps 11.5.4.1 to 11.5.4.8 for detectors 16-23.

11.4.3.10 Remove sources from detector drawers when counting is complete.

11.4.3.11 Review the IQC report for each detecctor.

11.4.3.12 Limits +-3% (fail)

11.4.3.12.1 Each analyst loading samples will verify that detectors are in service by reviewing the report in IQC for that day.

11.4.4 **Daily Source Criteria:**

11.4.4.1 Review and save with your name and date on the IQC report for each detector.

11.4.4.1.1 If a detector fails criteria, re-count source.

11.4.4.1.2 If detector fails source re-count tag detector with a Red out of service tag noted with date to indicate that the detector is out of service for the day

11.4.5 **Daily check Criteria:**

11.4.5.1 Review and save with your name and date on the IQC report.

11.4.5.1.1 The indiviuals loading samples will verify that detectors are in-service prior to loading on them.

11.4.5.1.2 In addition Daily checks will be verified at 1st level review of Data.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis ST-QAM.

12.3 To calculate the efficiency of the detectors for Cl-36, divide the net counts determined of the spiked Sodium Chloride, by the known dpm of the Standard used.

$$\frac{Net\ Counts\ of\ Spiked\ Silver\ Chloride}{Known\ dpm\ of\ Cl-36\,(decay\ corrected\ to\ day\ counted)} = Efficiency$$

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – (e.g. reprep/reanalysis, narration). If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL (or MDC when applicable) or greater than five times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed..

13.3.2.2 LCS Spike Recovery excursion (low) the batch is re-prepped and re-analyzed for the affected isotope.

13.4 RPD/RER Duplicate excursion – For the RPD/RER (or DER when requested by the client),one or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits (the DER is 3 or less depending on the significant digits). Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (i.e. nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narated in an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Carrier recovery must be within specified limits.

13.6.2 Carrier recovery low– Samples must be reextracted. Exceptions can be made and results reported with approval from the technical director, manager, or client and appropriate NCM included.

13.6.3 Carrier recovery high

13.6.3.1 A sample carrier recovery outside QC limits may be accepted if the sample results are determined valid by technical director, manager, and/or client approval:

13.6.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the

samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Radium	Strontium	Chloride
110%	107%	109%

13.6.5 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.7 Insufficient Sample

13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

16.2.1 The following waste streams are produced when this method is carried out.

16.2.1.1 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

17.1 ANSI N42.25-1997 – American National Standard Calibration and Usage of Alpha/Beta Proportional Counters

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay, Sr-02 Strontium 90, Sr-03-RC Strontium-90 in Environmental Samples.

17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry

17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes

17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium-228

17.6 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta

17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes

17.9 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium-228

17.10 Standard Method 7110C Coprecipitation Method for Gross Alpha Activity in Drinking Water, 19th Edition

17.11 TestAmerica St. Louis Quality Assurance Manual, current revision

17.12 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions

17.13 Associated SOPs, current revisions:

17.13.1 ST-PM-0002 "Sample Receipt and Chain of Custody"
17.13.2 ST-QA-0002, "Standards and Reagent Preparation."
17.13.3 ST-QA-0024, "Preventative Maintenance"
17.13.4 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"
17.13.5 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".
17.13.6 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
17.13.7 ST-RC-0021, "Gross Alpha Radition in Water using Copreciptation"
17.13.8 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"
17.13.9 ST-RC-0040, 'Total Alpha Emitting Isotopes of Radium"
17.13.10 ST-RC-0041, "Radium-226 and Radium-228 by Chemical Separation Preparation"
17.13.11 ST-RC-0050, "Aqueous and Soil Sample Preparation for Strontium-89, Strontium-90, and Total Strontium"

18.0 MODIFICATIONS TO THE REFERENCE METHOD

18.1 Strontium-89 short half life makes it impractical to use as a calibration standard for both radium-228 analysis, as stated in EPA method 904 and SW method 9310, and strontium-89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed strontium-90/yittrium-90 standard

for its' GFPC beta calibration used in Gross Beta, strontium-90, strontium-89, and radium-228 analyses. TestAmerica St. Louis has selected the strontium-90/yittrium-90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.

18.2 For Ra-228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.

18.3 TestAmerica St. Louis does not perform a direct strontium-89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total strontium and strontium-90.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated Section 10 to address voltage increase per step, plateau slope and QC check count requirements (5000 counts)

19.2 Rev. 11;
- 19.2.1 Added instument Purple throughout section 10 and 11.
- 19.2.2 Adjusted procedure steps throughout section 11.

19.3 Rev. 12,
- 19.3.1 Added Sr-02-RC and Sr-03-RC to sections 1.0 and 17.0.

19.4 Rev. 13:
- 19.4.1 Added Neptunium to scope in section 1.0.
- 19.4.2 Updated the Quality Control Program for counting daily rad checks and daily background checks in section 3.0.
- 19.4.3 Updated background count set-up, printing and entering protean data in section 10.8.

19.5 Rev. 14: (9/12/2013)
- 19.5.1 Removed references to Clouseau, SAC and QuantIMS
- 19.5.2 Section 5.0 added silver nitrate and ammonium hydroxide
- 19.5.3 Section 6.0 updated to include additional equipment
- 19.5.4 Section 7.0 updated to include addition reagents
- 19.5.5 Section 9.0 added reference to prep SOPs for additional information
- 19.5.6 Section 10.0 added sodium cloride standard preparation & reference to ST-QA-0024
- 19.5.7 Section 12.0 added Cl-36 detector efficiency calculation
- 19.5.8 Section 13.0 updated to include actual corrective actions and native concentration carrier requirements
- 19.5.9 Section 13.0 updated to include corrective actions
- 19.5.10 Section 17.0 added reference to ST-QA-0024

19.6 Rev. 15: (1/16/2015)
- 19.6.1 Added Section 7.10
- 19.6.2 Updated Section 9.6.1
- 19.6.3 Updated Section 10
- 19.6.4 Updated Section 11
- 19.6.5 Added ANSI N42.25-1997 reference to section 17

19.7 Rev. 16: (05/05/2015)
- 19.7.1 Section 11.3.3 added Background and checks are needed following a tank change.

19.8 Rev. 17: (05/23/2016)
- 19.8.1 Section 10, addition of trending of controls
- 19.8.2 Appendix created with trending rules

19.9 Revision 18 05/02/2017

19.9.1 Section 10.2 changes timefrom 5 minutes to "accumilate at least 10,000 counts" for distributed Sr90 source
19.9.2 Section 10.5 changed "single source" to "minimum of one source" for ICV/ACV
19.9.3 Section 10.9 – Added "Protean" source check instructions to those for "Orange" and "Purple" to combine all instruments. Added instrument "Blue" as well.
19.9.4 Section 11.3: added requirement to record gas tank changes on document on separate sheet
19.9.5 Updated SOPs in Section 17

19.10 Revision 19: (12/15/2017 (Tech Review: R. Mueller/J. Watson; QA Review: M. Ward))
19.10.1 Section 5: changed MSDS to SDS
19.10.2 Section 7:
19.10.2.1 Removed Ra-228 from beta calibration standard
19.10.2.2 Removed reference to Milli-Q system
19.10.3 Section 10.5.5 was removed to eliminate the need for ACV
19.10.4 Section 10.5.9 was revised to remove ACV
19.10.5 Section 18.0 removed- the lab changed the source for Ra-226 calibrations from Th-230 to Ra-226 and no note is needed to explain the use of a different analyte for calibration.

19.11 Revision 20: (1/16/2019)
19.11.1 Technical Review: Rachel Mueller/QA Review M Ward
19.11.2 Removed Neptunium calibrations throughout SOP- this is no longer determined by GFPC.
19.11.3 Fixed grammar, miss-types, and formatting throughout SOP.
19.11.4 Re-worded language throughout SOP to be more clear
19.11.5 Section 10.4: changed the language to describe the frequency of initial calibrations.
19.11.6 Section 10.4: moved cross talk section (sections 10.7 and 10.8) to the initial calibration subsection as this is determined during intial calibrations.
19.11.7 Section 10.4.5.4- added Co-precipitation calibration
19.11.8 Section 132.2.1: method blank criteria changed in accordance with QSM (5X)
19.11.9 Section 13.3.1- precision removed- mentioed in section 13.4
19.11.10 Section 13.4- added DER
19.11.11 Section 13.6: removed mention of tracer recovery and criteria- there are no current methods that use tracer recoveries for GFPC, the criteria mentioned is only meant for methods with tracers
19.11.12 Added reference to Standard Method 7110C to Sections 1 and 17

19.12 Added Eurofins logo and udpated copyright information (4/18/2019)

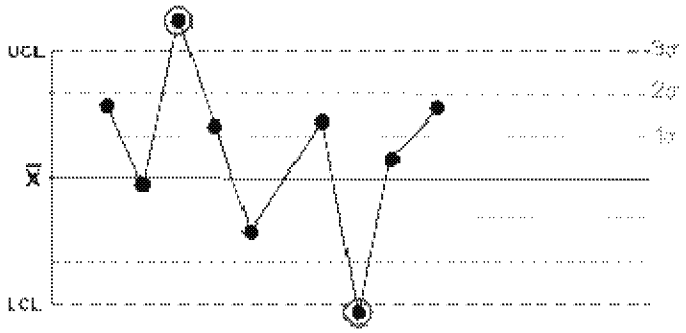
19.13 Annual Review (11/7/2019) Tech Review Jody Watson/QA Review Marti Ward
19.13.1 No Technical changes
19.13.2 Added new GFPC Instrument "Red" to Sections 10 and 11

Appendix

Rule 1: One point is more than 3 standard deviations from the mean.

~ Simplest: Single point is considered out of control event

- Corrective action required



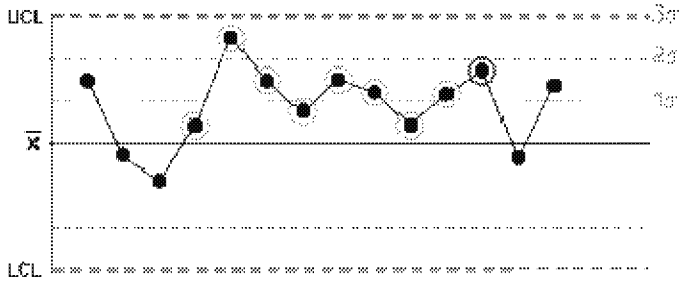
Rule 2: Seven (or defined 'n') points in a row are on the same side of the mean.

~ n = 7 warn trigger

- Mean shift: (same side of mean)

- Some prolonged bias exists.

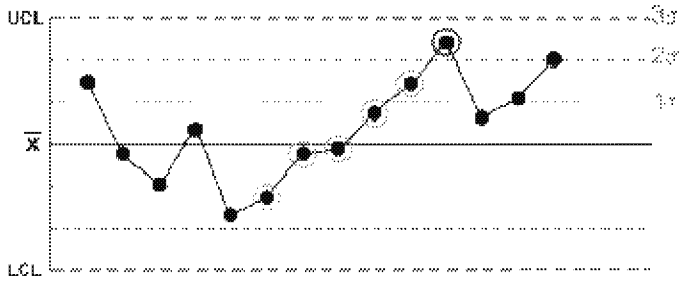
~ Corrective action recommended



Rule 3: Six (or more) points in a row are continually increasing (or decreasing).

- A trend exists.

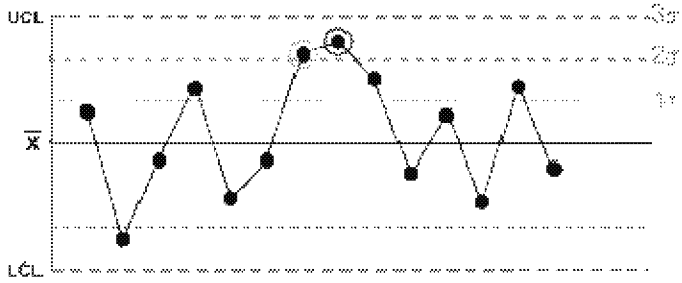
  ~ Corrective action recommended



Rule 5: Three out of three points in a row are more than 2 standard deviations from the mean in the same direction.

- There is a medium tendency for samples to be mediumly out of control.

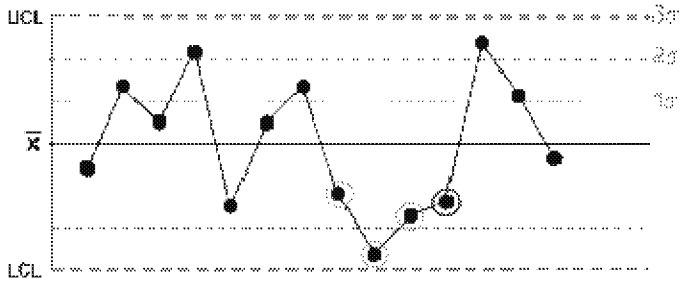
  ~ Investigate for preventive. May not be CAR



Rule 6: Four out of five points in a row are more than 1 standard deviation from the mean in the same direction.

- There is a strong tendency for samples to be slightly out of control.

  ~ No CAR, Statistical trend



CALIFORNIA
Water Boards
STATE WATER RESOURCES CONTROL BOARD
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE

ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

CERTIFICATE OF ENVIRONMENTAL ACCREDITATION

Is hereby granted to

NVL Laboratories, Inc.

PLM Dept.

4708 Aurora Avenue North

Seattle, WA 98103

Scope of the certificate is limited to the
"Fields of Testing"
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection, proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2757**

Expiration Date: **9/30/2021**

Effective Date: **10/1/2020**

Sacramento, California
subject to forfeiture or revocation

Christine Sotelo, Chief
Environmental Laboratory Accreditation Program

CALIFORNIA STATE
ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM
Accredited Fields of Testing

NVL Laboratories, Inc.
PLM Dept.
4708 Aurora Avenue North
Seattle, WA 98103
Phone: 2065470100

Certificate No. **2757**
Expiration Date **9/30/2021**

Primary Accreditation Body

Field of Testing: 121 - Bulk Asbestos Analysis of Hazardous Waste

121.010	001	Bulk Asbestos	EPA 600/M4-82-020	NVLAP

As of 10/1/2020 , this list supersedes all previous lists for this certificate number.
Customers: Please verify the current accreditation standing with the State.

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PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

NVL Laboratories

4708 Aurora Avenue North, Seattle, WA 98103

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2017 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 5.3 February 2018 and is accredited is accordance with the:

United States Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:

Environmental Testing

(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
April 6, 2012	February 12, 2020	May 31, 2022

Accreditation No.:	*Certificate No.:*
72200	L20-90

Tracy Szerszen
President/Operations Manager

Perry Johnson Laboratory
Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

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Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

NVL Laboratories

4708 Aurora Avenue North, Seattle, WA 98103

Contact name: Nghiep Vi Ly Phone: 206-547-0100

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Air	NIOSH 7300	ICP-AES	Arsenic
Air	NIOSH 7300	ICP-AES	Barium
Air	NIOSH 7300	ICP-AES	Cadmium
Air	NIOSH 7300	ICP-AES	Chromium
Air	NIOSH 7300	ICP-AES	Copper
Air	NIOSH 7300	ICP-AES	Lead
Air	NIOSH 7300	ICP-AES	Nickel
Air	NIOSH 7300	ICP-AES	Selenium
Air	NIOSH 7300	ICP-AES	Silver
Air	NIOSH 7300	ICP-AES	Zinc
Air	NIOSH 7400	PCM	Asbestos
Air	NIOSH 7082	FAA	Lead
Solid	EPA 6010D	ICP-AES	Arsenic
Solid	EPA 6010D	ICP-AES	Barium
Solid	EPA 6010D	ICP-AES	Cadmium
Solid	EPA 6010D	ICP-AES	Chromium
Solid	EPA 6010D	ICP-AES	Copper
Solid	EPA 6010D	ICP-AES	Lead
Solid	EPA 6010D	ICP-AES	Nickel
Solid	EPA 6010D	ICP-AES	Selenium
Solid	EPA 6010D	ICP-AES	Silver
Solid	EPA 6010D	ICP-AES	Zinc
Solid	EPA 7471B	CVAA	Mercury
Solid	EPA 600/M4-82/020	PLM	Asbestos
Solid	CARB M435	PLM	Asbestos
Solid	EPA 600/R-93/116	PLM	Asbestos
Solid	EPA 7000B	FAA	Lead

Matrix	Standard/Method	Technology	Analyte
Air	EPA 3051	Acid Digestion	Metals Digestion
Solid	EPA 3050B	Acid Digestion	Metals Digestion
Solid	EPA 1311	Leaching	TCLP

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF ASBESTOS CONTENT OF SERPENTINE AGGREGATE BY POLARIZED LIGHT MICROSCOPY and POINT COUNTING

SOP No.: 33.308.8
Revised Date: August 10, 2020
Author: Nick Ly

Approved by: Nick Ly, Technical Director

1.0 Scope

1.1 This method provides for the determination of Serpentine aggregate samples for the presence or absence of six types of asbestos: Chrysotile, Amosite, Crocidolite, Anthophyllite, Tremolite and Actinolite in accordance with guidelines promulgated by CARB Method 435 Implementation Guidance Document dated April 2017. This method also provides quantitative determination of asbestos content in accordance with section 8.3 of M435 point counting procedure.

2.0 Method Summary

2.1 A homogenized 100 grams of the original grab sample is removed and pulverized in a mortar and pestle prior to being sifted through a sieve with 200 mesh size (75 micrometers). The refined material is then examined under a low-power stereomicroscope for homogeneity, check for the presence or absence of fibrous constituents follow by preliminary fiber identification using a polarized light microscope (PLM). An asbestiform mineral is defined as having a minimum length to width aspect ratio of at least 3:1 (as defined by M435). Quantification of asbestiform mineral is performed by 400 point counting procedure.

3.0 Apparatus

3.1 Low-power stereomicroscope (10-45x) with light source
3.2 Polarized light microscope with wave retardation plate, 360 degrees graduated rotation stage, substage consenser, lamp and substage iris
3.3 Objective lenses: low power (10x), high power (40x); a medium power (20-25x) and very low power (2-5x) are optional
3.4 Dispersion staining objective lens (central stop)
3.5 Ocular lens (8x minimum)
3.6 Eyepiece reticule: cross hair
3.7 Compensator (wave retardation plate): 550 nanometer (1st order red or gypsum)
3.8 Mortar and pestle

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3.9 Metal sieve with a mesh size 200 (75 micrometers)
3.10 Forceps, scalpels, probes
3.11 Microscope slides
3.12 Cover slips
3.13 HEPA-filtered hood
3.14 Tissues and baby wipes
3.15 Glassine paper or clean glass plate
3.16 Index of refraction liquids ND = 1.490-1.720 in increments of 0.002 or 0.004.
3.17 Index of refraction liquids for dispersion staining: high dispersion series, ND = 1.550, 1.605 and 1.680
3.18 Hydrochloric acid
3.19 Organic solvents (THF, Acetone)
3.20 Drying oven

4.0 Sample Preparation

Prior to conducting analysis of a submitted sample, the original material is dried in a drying oven at approximately 100 Celsius to remove any moisture prior to thoroughly homogenizing the material in a sample container. A representative aliquot of 100 grams of the homogenized sample is removed and pulverized in a mortar and pestle to further refine the content prior to being screened through a sieve with 200 mesh size and transfer to a clean sample container. The refined material is then carried through the analytical procedure as specified in section 5.0.

5.0 Analytical Procedure

5.1 Pour a sample on glassine paper and examine under stereomicroscope that is contained in a HEPA-filtered hood. The sample prep hood must be left on when conducting sample preparation and analysis. Look for homogeneity, presence or absence of fibers and make a preliminary identification and estimate of fiber content using a PLM.

5.2 Place a drop of 1.550 refractive index liquid onto glass slide. Take pinches of representative materials and put them into the liquid. Tease apart or grind up materials into finer matrix using a metal probe. Cover with a cover slip. Make a minimum of three mounts per sample.

Remarks: Use 1.550 to check for Chrysotile; 1.680 for Amosite and Crocidolite; 1.605 for Tremolite, Actinolite and Anthophyllite.

5.3 Using a polarized light microscope, scan entire area, identifying asbestos, non-asbestos fibers and matrix. Note optical properties of asbestos fibers:
Refractive index (alpha and gamma) using central stop dispersion color in accordance with the method by Shu-Chun Su, sign of elongation, morphology, birefringence, extinction angle, color and pleochroism using plane polarized light, crossed polars and compensator plate for determination of characteristics.

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5.4 Note characteristics of asbestos and non-asbestos fibers and report at least one definitive characteristic of the predominant non-asbestos fiber that identifies it as non-asbestos fiber. Record all optical properties in the fields provided in the database. Identification of asbestos fibers can be verified by using the optical properties as provided by NIST Standard Reference Materials 1866a (Chrysotile, Amosite, Crocidolite) and 1867 (Anthophyllite, Tremolite and Actinolite). The accuracy of the asbestos fibers reported can be checked against the acceptable limits as established in the tables outlined in the NIST Certificate of Analysis.

5.5 Visually estimate percentages of asbestos and non-asbestos fiber content based on what was observed under both the stereoscope and PLM microscope. Compare this with prepared slides of known percentages and content of asbestos for accurate estimation.

5.6 Enter macroscopic description of sample, matrix materials, non-asbestos fiber type/percentage and asbestos type/percentage into data base.

5.7 All tools and equipment used around the stereoscope and work area must be wipe cleaned using a moist towel before preparing another sample for analysis. Sample slides, cover slips, disposable glassline paper, and dirty towels shall be disposed in a sealed Ziploc bag inside the sample preparation hood for later disposal.

6.0 Quantitation of Asbestos Content

6.1 Quantitation of the amount of asbestos present is most readily obtained by conducting 400 point counts on the prepared slides. Prepare 8 slides of approximately 5 milligrams of the refined material per sample to be used for asbestos identification and point counting. Count a minimum of 50 points per slide for a total of 400 points per asbestos mineral (LoQ of 0.25% or 0.3% based on 1 asbestos point per 400 total points counted).

6.2 If asbestos is consistently observed, but not counted, report <0.1% asbestos (LoD). Less than 0.1% (<0.1%) amount of asbestos is defined as having at least one asbestos fibers positively identified spreading over three or more sample slides and no point landed on an asbestos fiber during the point count analysis.

6.3 If no asbestos is observed in the sample preparation, report None Detected (0.0%) or No Asbestos Detected.

POINT COUNT PROCEDURE:

1. Sample movement by mechanical stage

   A. Mount prepared sample. Move along the X axis from one side of cover slip to the other. Start at upper left corner of sample.

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B. Use micrometer scale on mechanical stage to traverse sample in standard increments across slide. Movement should be standard and large enough to have a new field of view, relative to crosshair or reticle. If no mechanical stage is available on microscope, randomly move the sample slide across the stage.

C. Move standard increment on Y axis. Plan to do 8-10 traverses per slide.

2. Sample counting

A. At cross hair or reticle intersection, determine if space directly superimposed has fiber, matrix or empty space.

B. If fiber at crosshair, analyze by morphology and birefringence to confirm optical properties.

C. Record point as asbestos, matrix (include other fibers in matrix or count separately) or empty space. If an asbestos fiber & matrix particle overlap so that a point is superimposed on their visual intersection a point is scored for both categories.

D. Use a mechanical counter as record of count.

E. Move sample an X increment, observe material at crosshair. Repeat the process

F. Count a minimum of 400 non-empty points to determine whether a sample is greater than 0.25%, i.e. one asbestos point in 400 non-empty points. Count a maximum of 50 points per sample slide. Eight sample slides total.

Calculation of area percent asbestos:

%Asbestos = (# Asbestos/Total # non-empty points) * 100

7.0 Non-Asbestos Fibrous Components

1 Cellulose: Has refractive index similar to chrysotile. Cellulose exhibits pinch and swell characteristics along length, has internal cellular structure and lacks splayed ends. Fibers are not composed of bundles of smaller fibers.

2 Polyethylene fibers: Has morphology and refractive index similar to chrysotile, but will melt when exposed to heat. Has higher birefringence than chrysotile.

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3 Vermiculite: Has refractive index and extinction characteristics similar to chrysotile. May be colored green or brown and look more tabular than fibrous. No fibrils are present.

4 Fibrous talc: Has morphology and refractive index similar to chrysotile, but has a higher birefringence than chrysotile.

5 Glass fibers: Glass, while isotropic, may occasionally refract light in such a way as to mimic the morphology of amosite, but it will not show a sign of elongation under the compensator plate and the index of refraction will be different from amosite.

8.0 Documentation of raw data on optical bench sheet

8.1 All pertinent physical and optical characteristics for fibrous components must be documented on the optical bench sheet. These observations shall be recorded in the following order: sample color, sample texture, homogeneity, fiber morphology, % asbestos under stereo microscope, refractive index (alpha and gamma), fiber color under PLM, pleochroism, birefringence, extinction, sign of elongation, asbestos and non-asbestos fiber type, % asbestos under PLM and bench temperature.

9.0 Quality Control

9.1 Contamination check: Processing blanks such as powdered sugar or asbestos-free material shall be processed alongside regular field samples to check for cross-contamination on a daily basis.

9.2 Microscope Alignment and Refractive Index (RI) Liquid Calibration: Calibration of microscope shall be performed on a daily basis to ensure proper alignment of optics and operation of the microscope. Calibration of RI liquid shall be done when transferring from the stock to the working RI bottles or every 3 months in storage.

9.3 Sample QC duplicate re-analysis: QC duplicate re-analysis of field samples are conducted by a second analyst at a frequency of minimum 1 per 20 samples based on total samples analyzed by the first analyst.

9.4 NIST Standard Reference Materials 1866a (Chrysotile, Amosite, Crocidolite) and 1867 (Anthophyllite, Tremolite and Actinolite). The accuracy of the asbestos fibers reported can be checked against the acceptable limits as established in the tables outlined in the NIST Certificate of Analysis.

9.5 QC samples acceptance criteria

9.5.1 Sample Duplicate: Re-analysis of duplicate samples must have a precision of less than or equal to ($\leq$ 50%). When precision error is greater than 50%, the source of precision error in the sample duplicate

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must be investigated to determine homogenous nature of the sample and re-analysis of affected samples be carried out. If additional sample is not available, report findings in narrative.

9.5.2 Reference Sample : Analysis of reference samples must have an accuracy in the range of 50% to 150% recovery rate. When accuracy falls outside of the acceptance limits, the source of accuracy error in the reference sample must be investigated to determine the root cause and re-analysis of affected samples be carried out. If additional sample is not available, report findings in narrative.

10.0 Pollution Prevention

Samples shall be prepared in a clean well ventilated HEPA-filtered sample prep hood with minimal foot traffic or disturbance.

11.0 Waste Management

Samples shall be disposed of in accordance with federal, state and local regulations.

12.0 Equipment Maintenance

Daily and regularly maintenance of PLM using appropriate RI liquid and sample media to verify proper optical alignment and illumination.

13.0 Computer Software and Hardware

Data entry and tabulation are performed using Excel spreadsheet and/or Filemaker database to summarize data entry and generate test reports.

14.0 Troubleshooting

13.1 Check light source for burnt out bulb or fuse if microscope fails to light
13.2 Check and replace light socket or fuse if new bulb fails to light
13.3 Center stage and check optical alignment for proper illumination

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15.0 References

Determination of Asbestos Content of Serpentine Aggregate, Implementation Guidance Document, Field Sampling and Laboratory Practices, California Environmental Protection Agency Air Resources Board (CARB) Method 435, Updated April 2017.

Interim Method for the Determination of Asbestos in Bulk Insulation Samples, EPA Test Method 600/M4-82-020, 1982

Method for the Determination of Asbestos in Bulk Building Materials, EPA Test Method 600/R-93/116, July 1993

The Optical Microscopic Identification and Quantification of Bulk Asbestos, E.R. Crutcher.

A Rapid and Accurate Procedure for the Determination of Refractive Indices of Regulated Asbestos, Shu-Chun Su, Hercules Inc. Research Center, 1979-1982

Optical Mineralogy, Kerr, Paul F., 1977.

The Asbestos Particle Atlas, McCrone, Walter, 1980

Revision Record

Revision No.	Date	Parameters	Reviewer Initial
.2	01/10/2013	Added Waste Management and Troubleshooting	NL
.3	01/26/2016	Updated Item 12.0	NL
.4	01/20/2017	Updated Item 4.0	NL
.5	06/27/2017	Updated Analytical Procedure to comply with CARB 435 dated April 2017	NL
.6	07/23/2018	Clarification on mesh size number and particle size per CARB 435 dated April 2017	NL
.7	05/06/2019	Updated section 3.0 and 4.0	NL
.7	07/22/2020	Annual Review	NL
.8	08/10/2020	Updated section 5.1	NL

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Attachment 3

Field Sampling SOPs

(provided on electronic copy only)

- **ENV-601 Sample Handling and Custody Requirements**
- **ENV 603 Field Sample Documentation (Draft)**
- **ENV-610 Field Quality Control Samples**
- **ENV-801 Soil Sampling (Draft)**
- **ENV-802 Soil Characterization/Classification**
- **RP-104 Radiological Surveys (Swipe Samples / Section 10.3)**
- **RP 108 Count Rate Instruments**
- **RP 109 Dose Rate Instruments**

1.0 OBJECTIVE

The objective of the Sample Handling and Custody Requirements Standard Operating Procedure (SOP) is to establish procedures for the proper handling and custody of field samples.

2.0 BACKGROUND

Sample handling refers to how samples are labeled, packaged, shipped, and stored. Custody refers to how the samples are tracked and kept in possession by authorized parties (i.e. sample collectors, laboratory sample custodians, etc.), and how the transfer of sample possession is documented. Custodial possession is critical to show that samples have not been tampered with and remain representative of the site.

A sample is physical evidence collected from a site or facility. Sample handling and custody serves to maintain sample representativness (i.e. the degree to which samples reflect the matrix sampled), and serves as a means of tracking samples. Every sample will be given a unique field sample designation for identification purposes. Proper sample handling and custody maintains sample representativeness by controlling and preserving samples to prevent sample degradation and preventing cross-contamination by proper packing. Sample handling and custody entails selection of proper sample containers, sample preservation, shipping, sample custody, chain of custody, and sample packaging. .

Due to possible evidentiary nature of the samples collected during an investigation, a stringent program of custody procedures will be utilized to assure that each sample is accounted for from the time of collection to completion of analysis. To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory, documentation in logbooks and a Chain of Custody Record (COC) will be employed. COCs are generally provided by the laboratory, along with bottle order and coolers; however, an ECC COC may also be employed. An example COC is included at the end of this SOP. This COC or a similar one may be used should no others be available.

3.0 SAMPLE IDENTIFICATION

The method of sample identification used depends on the type of sample collected. Samples collected for specific analyses or measurement data will be recorded directly in bound field logbooks and recorded directly on the COC, with identifying information, while in the custody of the samplers.

All samples collected for shipping on a specific date will be recorded on the same COC, with the required analyses marked (one line per sample, with all required analyses for that sample marked on that line) unless sample quantities requires additional COCs or samples are to be shipped to more than one location (such as for specialty or radiological analyses). See the example COC at the end of this SOP. If additional instructions are needed or questions arise , contact the ECC Project Chemist for clarification.

All sample identification, COCs, receipts for sample forms, and field records will be recorded with waterproof, non-erasable ink. If errors are made in any of these documents, corrections will be made by crossing a single line though the error, entering the correct information, and initialing and dating the correction. If possible, all corrections will be made by the individual making the error.

If information is entered onto sample labels, logbooks, or sample containers using stick-on labels, the labels will not be capable of being removed without leaving obvious indications of the attempt. Labels will never be placed over previously recorded information. Corrections to information recorded on stick-on labels will be made as stated above.

Sampling locations will be numbered sequentially and coordinates, or at a minimum, a sketch, will be provided showing sample numbers and their corresponding locations. Every sample will be given a unique sample designation for identification purposes. The numbering system will be coordinated with the on-site representative to ensure that the proposed sample identifiers are discrete. Unless defined in project-specific

planning documents, the sample identification will be a series of letters and numbers consisting of three or four character strings, identifying the specific sample as follows:

PPPP-hhhh-xaaa

PPP is the 4 digit project code number (example: 5379)
"hhhh" is the unique identification assigned to each sampling location (or type for quality control [QC samples]) and depth.

Some examples:

EA= Excavation Area	SW= Source Water
DC= Drum Contents	EF= Effluent
DS= Debris Sampled	FD= Final Decontamination
IF= Imported Fill	EW=Excavation Water
PT= Post Treatment	WW= Waste Water
IL= Inline (Air Monitoring)	MS= Metals Contaminated Soil
OT= Operational Testing	FT= Water Samples from Frac Tank
DT= Demonstration Testing	UW= Upwind (Air Monitoring)
OT= Operational Testing	DW= Down Wind (Air Monitoring)
IW= Incineration Waste	SC= School Zone
WZ= Work Zone	

EB= Equipment Blank
FB= Field Blank
FD= Field Duplicate
FS= Field Spike
TB= Trip Blank

The second "hh" will be used to indicate sample areas or other identifying designation. Specific notations regarding the meaning of the identifiers will be made in the logbook.

"x" describes the sample matrix:

1= soil
2= water/liquid
3= debris
4= air

For example: Sample No. 5379-EA01-1001 indicates that the sample is:

For Project No. 5379
Collected at the excavation area 01; and is
Soil sample 001.

All logbooks, sample labels/tags, custody seals, representative sampling documents, and COCs will be completed using the same sample designations.
See also SOP ENV-603 Field Sample Documentation.

4.0 SAMPLE PRESERVATION

Sample preservation ensures the samples are representative of the matrix sampled, by preventing sample degradation, volatilization, or precipitation. Samples are preserved by addition of chemical preservatives to the sample and/or by cooling the samples to 4 degrees Centigrade (°C) ± 2°C immediately upon sample collection. Clean containers ensure samples are not cross-contaminated. Sample containers and preservation required, along with contact information to place a bottle order with the laboratory, will be available in the site-specific Sampling and Analysis Plan (SAP) (possibly identified as the Uniform Federal Policy – Quality Assurance Project Plan [UFP-QAPP]).

Certified, commercially clean sample containers will be used for submitting samples for analysis. Sample jars or bottles should have a certificate of analysis from the manufacturer the bottles or jars, which show the bottles or jars, are contaminant free. Sample containers containing the correct preservative are available from the laboratory (see below); however, bottles or jars with preservatives added at the laboratory may lack these certificates. Check with the laboratory to determine that preservatives were added to certified commercially clean containers.

Typically, chemical preservatives are added to the sample bottle by the contract laboratory before the bottles are shipped to the site, and the sample bottles should indicate the type of added preservative. Contact the laboratory for more containers if the containers are not preserved or if preservatives must be added in the field. Check the preservative levels in the bottles at least once during the beginning of the project. Determine if the preservative pH is within required limits by sample preservation as specified in the SAP. Some sample matrices may require additional preservative. If the pH is not in the proper range, notify the contract laboratory.

Samples will be packed in ice as noted in Section 10.0; samples that are hand-delivered to the laboratory on the day of sample-collection may not need to be iced.

5.0 SAMPLE LABELS

Sample labels are used to identify samples collected for laboratory analysis. A sample label will be securely attached to each sample container. Some labels may not adhere to chilled sample bottles. For non-volatile analyses only, clear tape may be used to secure the labels in place. In some cases, particularly with biological samples, the sample labels may have to be included with or wrapped around the samples. The sample labels are sequentially numbered and are accountable documents after they are completed and attached to a sample or other physical evidence. The following information, at a minimum, will be included on the sample label using waterproof, non-erasable ink:

- Project number;
- Unique sample number and sample location;
- Date and time of sample collection;
- Sample description and location;
- Designation of the sample as a grab or composite;
- The signature of either the sampler(s) or the designated sampling team leader and the field sample custodian (if appropriate);
- Whether the sample is preserved or unpreserved; and
- The general types of analyses to be performed.

6.0 CUSTODY SEAL

After collection, separation, identification, and preservation, the sample will be maintained under chain-of-custody procedures. Due to the evidentiary nature of the samples collected, possession must be traceable from the time the samples are collected until valid analytical results are obtained. The custody seal will be attached to the outside of each sample container in such a manner that it is necessary to break it in order to open the container. The following information, at a minimum, will be written on the custody seal:

- Date; and
- Signature of sampler(s)

Containers for samples to be analyzed for volatile organic compounds (VOCs) must not be taped other than with the standard custody seal. Custody must, at a minimum, be placed upon the sample coolers in the manner described.

7.0 SAMPLE CUSTODY

A sample is considered under custody if any one of the following conditions is met:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in his or her physical possession;
- It was in the physical possession of an investigator who then secured it to prevent tampering; and/or
- It is placed in a designated secure area with limited or controlled access.

8.0 CHAIN-OF-CUSTODY RECORDS

All sample shipments to on-site or off-site laboratories will be accompanied by the COC identifying its contents. The original COC will be shipped along with the samples, while the person who initiates the record (the originator) will retain a copy. Electronic COCs may be transmitted to the laboratory in advance of sample receipt. COC procedures are comprised of the following elements:

1. Maintaining sample custody and
2. Documentation of samples for evidence.

The COC ensures that samples are traced from the time of field collection until valid analytical results have been obtained.

When responsibility for group of samples changes several times, each custodian is not required to retain a copy of the COC, as long as the original COC indicates that each person accepting the samples has

subsequently relinquished custody appropriately. COCs will be completed according to the following protocol:

- The originator will fill in all requested information from the sample labels;
- The originator will sign and date the "Relinquished by" box and keep the copy;
- The Federal Express Tracking number or other reference shipping number, if any, will be included in the sample shipment documents;
- The original COC will be shipped with the samples. A plastic shipping envelope will be taped to the inside of the cooler top and the COC will be inserted;
- A copy of the COC will be retained by project personnel.
- The person receiving custody of the sample at the laboratory will check the sample label information and the COC, and will also check sample condition and note unusual observations on the sample log-in sheet;
- The person receiving custody will sign in the adjacent "Received by" box and keep the original;
- When samples are shipped via common carrier (e.g., Fed Ex), the date/time will not be the same for both signatures;
- When samples are shipped via common carrier, the original COC will be shipped with the samples and the shipper (e.g., Field Sample Custodian) will keep the copy.
- The shipper will also keep all shipping paper, bills of lading, etc;
- In all cases, it must be readily apparent on the COC that the person releasing custody has relinquished it to the next custodian; and
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the COC.

9.0 REPRESENTATIVE SAMPLE DOCUMENT

A representative sample document will be completed for each sample collected. This will, at a minimum, be the field logbook; however, clients may require documentation in addition to the field logbook, in which case all required client documents will also be completed.

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The representative sample document(s) will record the complete history of each sample, including the sample source, date collected, sample methodology, sample size and container, sample number, and the analysis required. This document will be signed by the sampler and a witness, and will remain with the project records.

10.0 SAMPLE HANDLING AND SHIPPING

Place samples into appropriately labeled and preserved sample containers and immediately store on ice at 4 ± 2 °C in an insulated cooler. Keep cooler under observation and in a shaded area and maintain custody over the samples. Document samples that are collected in the field logbook. (Samples to be hand-delivered to the laboratory on the same date as collection may not require ice, and may not have reached 4°C ± 2°C even if iced. It is important to note hand-delivery on the COC and field logbook).

Wipe excess soil or water off of the sample bottles or jars before storing or shipping the samples. Wear appropriate personal protective equipment (PPE) as described in the Health and Safety Plan while handling samples.

Prior to shipping the samples, prepare the COC. Ensure the COC and sample label information agree and that the COC accurately indicates all of the samples in the cooler. Place the samples in Ziploc storage bags and wrap in bubble wrap. Place the completed COC in a plastic bag and tape the COC to the inside top of the cooler used for shipping. Keep a copy of the COC in the field records.

Prior to placing samples in the cooler, place 3 inches of inert packing material (such as waterproof Styrofoam "peanuts") on the bottom of the cooler. Place samples upright into the cooler, and do not let the samples touch each other. Add a temperature blank to the sample cooler. The laboratory measures the temperature of the temperature blank to determine and document the temperature of the samples upon receipt at the laboratory.

Place ice (double-bagged in Ziploc bags) around the samples and on top of the samples. Fill in voids with packing material or bagged ice, but do not over pack too tightly. Tape shut the cooler drain port. Use strapping tape to secure the cooler lid. Place two custody seals on the cooler – one on the front and one on the back and initial and date the custody seals in such a manner that the seals will break if the cooler is opened. If the sample custodian is to hand-deliver the samples to the laboratory, and the sample coolers are in the control of the custodian at all times prior to delivery, custody seals on the cooler are not needed.

Ensure samples reach the analytical laboratory and are analyzed well before the holding time expires.

Notify the laboratory of sample shipments on the weekend to ensure a sample custodian is present to receive the samples, and notify the laboratory if the number of samples per day or type of analysis is greater than that specified in the laboratory contract.

11.0 REFERENCES

Air Force Center for Environmental Excellence (AFCEE), 2004. Guidance for Contract Deliverables, Appendix C: Quality Assurance Project Plan, July 2004.

Department of Defense (DoD), 2009. DoD Quality Systems Manual for Environmental Laboratories, April 2009.

Department of the Navy, 2009. Navy Environmental Compliance Sampling and Field Testing Procedures Manual, Revision 1, August 2009.

United States Army Corps of Engineers (USACE) 2001, Engineering Manual (EM) 200-1-3, February 2001.

United States Environmental Protection Agency (EPA), 2007, Contract Laboratory Program Guidance for Field Samplers, July 2007.

Chain-of-Custody Forms

/Genr/SOPDB/Files//SOP 601 COC + example COC.pdf

1.0 PURPOSE

The purpose of the Field Documentation Standard Operating Procedure (SOP) is to describe the procedures for maintaining sample control through proper sample documentation. When samples are collected for chemical or physical characteristic analysis, proper documentation needs to be completed. The information presented in this SOP enables maintenance of sample integrity from time of collection through transportation and storage. It is this documentation that will verify that the samples were properly handled.

1.1 Documentation

The following discussion outlines standard practices and procedures to be used when documenting a sampling episode. All project-specific documentation requirements must be presented in the Sampling and Analysis Plan (SAP). This includes identification of procedures required for field documentation, sample labeling, and the maintenance of chain-of-custody (COC). For United States Corps of Engineer's (USACE) projects, the Contractor is required to obtain a tracking number (e.g., Laboratory Information Management System (LIMS) number) to be used in conjunction with the government quality assurance (QA) sample shipments. The tracking number should be specified in the SAP. Proper completion of the logbook and supporting paperwork with indelible ink is necessary to support potential enforcement actions that may result from the sample analysis. Therefore, maintaining sample integrity through proper documentation is essential.

1.1.1 Field Logbooks

A field logbook, or electronic field notes (COCs and collections logs), may be used to record data collection activities performed at the site. If electronic notes are recorded, field logbooks will serve as a backup. Logbooks will be bound and contain consecutively numbered, water-resistant pages. The first page of each logbook will contain the project number. All logbook entries will be in indelible ink. Notations should be made in logbook fashion, noting the time and date of all entries. All corrections will be made using a single lineout deletions and will be initialed by the person making the correction. At the end of each field day, the end of the last page used will be initialed and dated by the author(s), and a line will be drawn through the remainder of the page. The cover of each logbook will contain the project name and number, log start date, and log end date. A combination of the entries to the logbook, electronic forms, and referenced hardcopy forms will be sufficient to recreate the activities of the day and must include at least the following information:

- Name and title of author;
- Name(s) and title(s) of the field crew;
- Date and time the field work started;
- Location and description of the work area;
- Field instrumentation and equipment used and purpose of use (i.e., health & safety screening, sample selection for laboratory analysis), calibration methods used, field results, and quality control (QC) information;
- Meteorological conditions at the initiation of fieldwork and changes during the sampling event; and
- Personal and equipment decontamination activities.

For each sample collected, the following information must be recorded:

- Type of sample;
- Date and time of sample collection;
- Sample collection or measurement method;
- Sequential number and volume of sample taken;
- Location and description of sampling point;
- Sample identification number;
- Sample containers (size/type) and preservatives used in sample;
- Sample manipulations (i.e., filtration, compositing, etc.);
- Destination of samples after collection;
- Description of the number of shipping coolers packaged with corresponding COC numbers and shipping method employed (noted applicable tracking numbers);
- Name and address of all receiving laboratories (include LIMS number for QA laboratory);
- Reference entries for all maps and photographs of sampling site;
- Field observations/comments;
- Field measurements taken (pH, temperature, etc.);

- Serial numbers for field instruments;
- Time and date of last calibration of field instruments; and
- Any deviations from the SAP.

No field logbooks will be destroyed or thrown away, even if illegible or containing inaccuracies requiring a replacement document. Replacement documents will be so noted and referred to the original.

2.0 DOCUMENTING SAMPLING POINTS

The exact locations of sampling points should be documented for purposes of generating an accurate representation of the site conditions using the data generated to date, defining data gaps, and identifying potential future data needs. This is accomplished through the use of a monument and compass. A monument should be chosen at each site to act as a stationary reference point from which all sampling points can be measured using a compass and measuring tape. If a building or other stationary structure exists, its corner may act as this reference point. If no monument exists, it will be necessary to create one using a piece of wood, approximately 2 inch by 2 inches, hammered into the ground almost to ground level. The stake should then be marked with flagging tape or fluorescent paint. When establishing a sampling point, the following procedure should be used:

- Standing at the monument, facing the sampling point, use the compass hairlines to determine the degree of direction;
- Ensure that the line of sight runs from the monument, through both hairline needles on the compass, to the sampling point; and
- When first establishing the sampling point, record in the field logbook the degree and compass directional reading, along with the distance measurement from the monument to the sampling point, sampling stake or surveyed pin flags.

3.0 PHOTOGRAPHS and VIDEOS

All sampling points should be documented by photographs to allow for positive identification of the sampling points. Photographs are the most accurate and convenient records of field personnel observations. Photographs taken to document sampling points should include two or more reference points to facilitate relocating the point at a later date. Keeping a record of photographs taken is crucial to their validity as a representation of an existing situation. Color photographs will be taken of the field activities using either electronic camera or a film camera-lens system with a perspective similar to the naked eye; telephoto or wide-angle shots can not be used for enforcement purposes. The electronic source will be stored in the project files, or photocopies of hard copies and or negatives, will retained be made of the photographs as required for reporting purposes. Electronic photographs will be loaded the project sharedrive (or equivalent), into a folder designated for the day the samples were collected. If possible photographs or videos will taken on smart phones or tablets. The following information concerning each photograph will be recorded in either combinations of the field notebook, a photo log, or in the electronic cameras meta data file:

- Photographer's name;
- Name of Site *;
- Date and time of the photograph *;
- General direction the camera was facing *;
- GPS Coordinates *;
- Altitude *;
- Brief description of the subject and field work portrayed *;
- Sequential number of the photograph as indicated on the camera counter (if applicable);
- Site photo map (if applicable)*; and
- The film roll number on which the photograph appears (if applicable).
- Optional:
    - KML File generation for mapping
    -

* Camera applications utilized by smart phones are available that collect and store the information to reproduce the conditions and locations of the photo [Example: Solocator - GPS Field Camera App]

4.0 SAMPLE COLLECTION PAPERWORK

4.1 Sample Labels

Sample labels are required for the proper identification of the samples. All samples must be properly labeled with the label affixed to the container prior to transportation to the laboratory. It is also recommended that samples be photographed so the labels are clearly legible for later identification. Information on sample labels should include, but not be limited to, the following:

- Project Number, Code, or Name;

- Sample Identification Number *;
- Barcode / QR Code (if required);

- Sampler's name and signature or initials;
- Preservatives used (if required)*;
- Analysis/Analyses requested*;
- Date & Time of sample collection;
- Type of sample (discrete or composite)*; and
- Optional:
  - QC Designation (MS/MSD)
  - Federal Express tracking number (recorded on chain of custody).

** Refer to QAPP/SAP for details, if one has been prepared.*

4.2 Sample Numbering System

A sample numbering system should be used to identify each sample collected and submitted for analysis. The purpose of the numbering system is to assist in the tracking of samples and to facilitate retrieval of analytical results. The sample identification numbers for each sampling effort should be used on sample labels, sample tracking matrix forms, COC forms, field logbooks, and all other applicable documentation. A listing of all sample identification numbers should be recorded in the field logbook. The sample numbering system may vary depending on the number and type of samples that will be collected at the site.
Each sample collected must be assigned a unique sample number. Sample numbers should change when the media or location changes. However, sample numbers should not change because different types of analysis are requested.

Sample ID's may contain the following:

- Project Number or Project Name Acronym
- Matrix of the sample
- Sub-Location within project site (building name, grid ID,MW ID, etc)
- Sample Date
- Unique Sample Number
- Field QC Designation (eg. D= Field Duplicate, T=Trip Blank, etc)

4.3 Chain-of-Custody Record

All sample shipments will be accompanied by the chain-of-custody record (Appendix B) identifying its contents. This record will be used to document sample custody transfer from the sampler, to other sampling team members (if necessary), to the courier, and finally to the analytical laboratory. The chain-of-custody record ensures that samples can be traced from the time of field collection until they are received and analyzed by the analytical laboratory. The original custody record is shipped along with the samples, while the initiator of the record retains a copy. The information required for the chain-of-custody record includes:

- Type of sample (grab or composite) and matrix;
- Analytical method numbers and parameter names;
- Sample number;
- Signature of sampler;
- Date and time of sample collection;
- Project name, location and address; and
- Signatures of persons involved in the chain of possession.

When responsibility for a group of samples changes several times, each custodian is not required to retain a copy of the chain-of-custody record, as long as the original custody record indicates that each person accepting the samples has subsequently relinquished custody appropriately. chain-of-custody forms will be completed according to the following protocol:

- The originator fills in all requested information from the sample labels;
- The originator signs the "Relinquished by" box and keeps the copy;
- The original record sheet is shipped with the samples. A plastic shipping envelope will be taped to the inside of the cooler top and the remaining two copies of the chain-of-custody record will be filed with the representative sampling documents;
- The person receiving custody checks the sample label information against the custody record. He/she also checks sample condition and notes anything unusual under "Remarks" on the custody form;
- The person receiving custody signs in the adjacent "Received by" box and keeps the original;
- The Date/Time will be the same for both signatures, since custody must be transferred between two individuals.

However, when samples are shipped via common carrier (e.g., Federal Express), the date/time will not be the same for both signatures;

- When samples are shipped via common carrier, the original custody form is shipped with the samples and the shipper (e.g. Field Sample Custodian) keeps the copy. The shipper also keeps all shipping paper, bills of lading, etc.;
- In all cases, it must be readily seen that the person receiving custody has relinquished it to the next custodian; and
- If samples are left unattended or a person refuses to sign, this must be documented and explained on the chain-of-custody record.

In addition to the COC record, a COC seal is used to ensure that no sample tampering occurred between the field and the laboratory analysis. The COC seal is an adhesive seal placed in areas such that if a sealed container were opened, the seal would be broken.

1.0 OBJECTIVE

The objective of this Field Quality Control Sample Standard Operating Procedure (SOP) is to establish procedures for the preparation and implementation of field quality control samples.

2.0 BACKGROUND

Field quality control samples are prepared as a check of field sample collection methods and as a check of cross-contamination of sample in the field or during shipment. Equipment Rinsate, Field Blank, and Trip Blank samples check for potential cross-contamination, which impacts sample representativness. Field duplicates measure the precision and reproducibility of the field sampling efforts. Matrix Spike/Matrix Spike Duplicate (MS/MSDs) samples determine the potential for site matrix interferences in determining the true level of contaminants in the sampled matrix. Field quality control samples consist of the following:

Field Duplicate/Replicate samples are independent samples of a homogeneous matrix collected at the same time from the same location and are analyzed to check the field variability of sampling. Field Duplicates are generally required QC samples according to many analytical methods.

Equipment Rinsate samples are samples of analyte free (deionized) water which are rinsed over decontaminated equipment, collected, and submitted for analysis. Equipment rinsate samples are analyzed to determine if contaminants from one sampling activity are being transferred to another sample. The primary purpose of running Equipment Rinsate samples is to determine if the decontamination procedures are adequate.

Field Blanks are samples that are prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example: air-borne dust or organic vapors).

Trip Blanks are required only when samples are collected for analysis of volatile organic compounds (VOC)s. Trip Blank samples are prepared prior to the sampling event in the actual sample container and stored continuously with the investigative samples throughout the sampling event. Trip Blanks are used to determine if VOC samples were contaminated during storage and/or transportation back to the laboratory (a measure of sample handling variability resulting in positive bias in contaminant concentration). Trip Blanks are collected as a quality control check on aqueous VOC samples submitted to the laboratory, and to determine if the aqueous VOC samples were contaminated by either sample containers or during sample shipment.

Matrix Spike/Matrix Spike Duplicates are samples collected from the site and are used by the laboratory to determine the potential for matrix interferences, which may bias results. Matrix interferences are site specific, so site samples should be obtained for MS/MSD samples. Some laboratories will use soils or waters from an environmental sample for the MS/MSD sample. MS/MSD samples, whether collected as samples or obtained from environmental samples, are generally required QC samples according to many analytical methods.

3.0 EQUIPMENT REQUIRED

- Labels;
- Custody seals;
- Chain-of-Custody forms;
- Appropriate sampling containers;
- Indelible ink pens; and
- Field book.

4.0 PROCEDURE

Field QC samples shall be collected, containerized, preserved, stored, and shipped in the same manner as environmental samples. Generally, MS/MSDs, Equipment Rinsates, Field Blanks, and Trip Blanks are designated on the Chain of Custody and sample designation, as prescribed in the Field Sampling Plan or as in SOP –102 Sampling Handling and Custody Requirement.

4.1 Sample Handling and Mixing

After collection, all sample handling should be minimized. Investigators should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, investigators should ensure that the sample containers do not become submerged in melted ice, as this may result in sample cross-contamination. Plastic bags, such as Zip-Lock® bags or similar plastic bags sealed with tape, should be used when small sample containers (e.g., VOC vials or bacterial samples) are placed in ice chests to prevent cross-contamination.

It is extremely important that soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually;
2. Two quarters should then be mixed to form halves; and
3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4.2 Field Duplicate/Replicate Samples

Field Duplicate/Replicate samples will be collected at the same sampling location and immediately placed into the proper sample containers. Each duplicate sample will be analyzed for the same suite of analytes as the original sample. A minimum of one duplicate will be collected per twenty samples, but no less than one duplicate per sampling shift.

Duplicate samples will be collected by initially collecting a minimum of twice as much material as normally collected for a sample. After mixing, the material will be apportioned into two sets of sample containers. Both sets of containers will be submitted for analyses with one set designated with an appropriate sample identification number; the second possessing a fictitious sample number to mask the identity of the duplicate sample from the laboratory.

4.3 Equipment Rinsate Samples

Rinsate samples are samples of deionized (DI) water poured over decontaminated sampling equipment. The Rinsate Blank is collected during the final rinse of the sampling equipment after decontamination procedures are performed. Rinsate samples are analyzed for the same parameters as field samples to evaluate the effectiveness of field decontamination procedures. One rinsate Blank will be collected each day that sampling is performed.

4.4 Field Blanks

Field Blanks are collected in the field during sampling activities by pouring analyte-free water directly into the sample containers at the predetermined field sampling location. Field Blanks are analyzed for the same analytes as the related samples. At a minimum, one Field Blank will be collected during each sampling day. Field Blanks should be collected in dusty environments and/or in areas where volatile organic contamination is present in the atmosphere that originates from a source other than the source being sampled. Field Blanks are analyzed for the same analytes as the related samples. Field Blank samples will be collected with no headspace present within the sample container.

4.5 Trip Blanks

Trip Blanks generally are collected as a quality control check on volatile organic samples. When volatile organic samples are shipped to the recipient laboratory, trip Blanks will be provided with each shipment, but not for each cooler. They are packaged for shipment with the other samples and submitted for analysis. At no time after their preparation are trip Blanks to be opened before they reach the laboratory. Trip Blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample tags and Chain-of-Custody Records as Trip Blanks. A minimum of one Trip Blank shall be transported per one to ten samples collected.

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site:

Water Sample VOC Trip Blank - A water sample VOC trip Blank is required for every study where water samples are collected for VOC analysis. Two sealed preserved (or unpreserved if appropriate) 40-ml VOC vials will be transported to the field. For routine studies the samples will be prepared by lab personnel. Investigators shall request that these samples be provided at least one week in advance of scheduled field investigations and inspections and never less than two days in advance of scheduled field investigations and inspections (except in emergency situations). These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation.

Soil Sample VOC Trip Blank - A soil sample VOC trip Blank is required for every study where soil samples are collected for VOC analysis. The preparation and pick up of this sample will be the same as for the water sample VOC trip Blank. One two-ounce soil VOC vial will be transported to the field. This Field Blank will be handled and treated by personnel in the same manner as the soil samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample

tags and Chain-Of-Custody Records as trip Blanks.

- MS/MSDs - MS/MSDs are collected from the same sample interval or location as environmental samples. The MS/MSDs are required batch QC samples. If MS/MSD samples are not sent samples volume from environmental samples from the site or another site will be analyzed by the laboratory.

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to discuss equipment and appropriate collection methods for soil sampling. This SOP also provides general sampling instructions for composite sampling and the sampling of specific volatile organic compound (VOC) analytes.

2.0 BACKGROUND

Prior to conducting a soil sampling investigation, a sampling strategy will be developed based on the objectives of the investigation. After designing a soil sampling strategy, the appropriate equipment and techniques must be used to conduct the investigation. This SOP details the following soil sampling methods:

- Surface soil sampling;
- Sampling of soil wastepiles;
- Sampling soil from backhoes;
- Sampling soil from excavations;
- Split-spoon and direct push sampling for collection of soil samples at targeted depths below surface;
- Continuous core auger sampling; and
- Bulk sample collection for large soil sample quantities.

The techniques employed to obtain a soil sample depend on the physical properties of the material to be sampled, the depth from which the sample is collected, the analyte, the quantity, and the location to be sampled. Manual techniques and equipment, such as stainless steel scoops/spoons or hand augers, are usually used for surface or shallow subsurface soil sampling. Power operated equipment is usually associated with collecting deep samples. Power equipment can be used for collecting shallow samples in the event the auger hole begins to collapse, or if soil is so resistant that manual augering is not practical. Samples may be either grab or composite samples depending upon the project sampling objectives. Additional factors to consider during sample collection procedures are the time required to fill a sample container after exposure of the soil to the atmosphere, and the degree of acceptable soil disturbance.

3.0 EQUIPMENT REQUIRED

A copy of the Sampling and Analysis Plan shall be available for review at the site. The following is a list of equipment that may be used for surface soil sampling:

- Stainless steel scoop or spoon or Stainless steel hand-trowel;
- Stainless steel mixing bowl;
- Sample location stakes or flags (survey flags, PVC, wooden, etc);
- Back hoe;
- Disposable gloves;
- Field Notebook
- Chain of Custody
- Tablet / Electronic Field Data Recorder
- Sample description / Field collection Forms;
- Camera (and film, if required) *;
- Pens, pencils,and indelible markers;
- Labels;
- Clinometer *;
- Global positioning system (GPS) *;
- Slide Hammer – Brass sleeves for VOC sample collection;
- Shovels;
- Post-hole hand auger with a 5 foot extension;
- Casing;
- Polyethylene sample bags with fasteners (Ziploc type);
- Sample containers (Refer to SAP, field manual (if in field analysis), selected lab, or project chemist for proper sample containers)
- Plastic wrap and clean plastic sheeting;

- Cooler and ice packs (for samples);
- Vermiculite packing material;
- Triplicate Chain-of-Custody forms;
- Representative sample documents;
- Custody seals;
- 55-gallon drums with ring-type closures;
- PID with a lamp with an Ev setting as specified in the Health and Safety Plan;
- Appropriate safety equipment (Heat Stress Monitoring, CGI, PID, etc.) and PPE as specified in the Health and Safety Plan; and
- First aid kit.

** these items are available via smart phone apps.*

In the event that soil VOC samples are to be obtained, the following equipment may be utilized:

- Soil VOC samples by Method 5035 – top loading balance accurate to 0.01 grams and calibration weights; and
- Soil VOC samplers by Method 5035 - EnCore tm or Purge and Trap Soil Sampler tm Devices.

4.0 SAMPLING PROCEDURES

The two primary types of surface and subsurface samples are composite and grab samples. The samples are defined on the basis of their sampling origin:

Grab Sample – an individual sample collected from a single location at a specific time or period of time.

Composite Sample – a sample collected over a temporal or spacial range that typically consists of a series of discrete, equal samples (or "aliquots") that are combined or "composited".

4.1 Surface Soil Sampling

Sample locations shall be marked by a stake and surveyed or otherwise recorded with reference to local landmarks in such a manner that the location can be re-located.

4.1.1 Surface Soil Sampling Procedures

All sampling will be performed with properly decontaminated tools and equipment. Samples will be placed into properly labeled sample jars or vials. The proper field book and chain-of-custody entries will be made immediately after sample collection, and the sample will be placed on ice in a cooler chest at a temperature of approximately 4°C. Proper custody of the samples shall always be maintained. All sampling equipment will be decontaminated in accordance with standard decontamination procedures before moving to another sample location.

Non-VOC grab samples

Remove debris, grass, roots, rocks or other material leaving only the bare soil. When sampling from a backhoe bucket or a spoil pile, remove the upper most layer of soil, as this soil may consist of nonrepresentative adjacent soil layers. Prior to entering an excavation to sample the sidewalls, determine if the excavation presents a confined space or collapse hazard. For confined space or other excavations with forbidden entry, sample from the surface using a slide hammer. When using the slide hammer to sample an excavation sidewall, discard the upper quarter inch of soil, as this soil may be smeared from adjacent soils. If entry into the excavation is possible, other sampling instruments (hand trowels, spoons, etc.) may be used, once the sidewall surface has been scraped to the original matrix. Use a stainless steel spoon, slide-hammer, hand trowel, or hand auger to collect a sample from the depth specified in the Field Sampling Plan. Collect the soil from the specified depth and place the soil in a stainless steel mixing bowl. Avoid collecting oversized rocks, roots, or other non-soil debris unless specified in the Field Sampling Plan. Collect enough soil volume from the desired depth to fill the required sample jar(s). Homogenize the soil in the mixing bowl, by the quartering method – divide the sample into quarters – mix each quarter thoroughly, and then remix all four quarters. From the homogenized soil, fill the sample jars as full as possible.

Non-VOC Composite Samples

Perform the sample collection procedures as described above for non-VOC grab samples. Obtain the required number of discrete samples from various depths in an excavation, locations in an area, or sidewall locations, as described in the Field Sampling Plan. A composite sample shall be comprised of not more than 8 discrete samples.

4.2 Bulk Soil Sample Collection

Bulk samples consist of large volume samples from shallow pits generally collected using a backhoe, and loaded into 55-gallon drums. Prior to sampling, all equipment will be decontaminated in accordance with standard decontamination procedures.

First scrape off the upper few inches of the area to be sampled with the backhoe and place the material to the side. Decontaminate the backhoe bucket. Place a suitable number of 55-gallon drums (estimate 500 pounds per drum)

onto pallets near the backhoe for filling. After each drum is filled, seal it with the lid and ring closure. Label each drum with an appropriate sampling label with the following information:

- Date;
- Project;
- Location;
- Contents;
- Point of Contact Telephone number;
- Drum Tracking Number or Sample Designation;
- Depth; and
- Number of drum in total (Example: 1 of 3; 2 of 3; 3 of 3).

Before moving the filled drums, clean off any material spills that may have occurred during the sampling process.

4.3 Subsurface Soil Sampling

The method used for subsurface soil sampling is generally a function of the depth of the sample. The interval to be sampled may be determined by the results of PID or Headspace Analysis.
However, once the sample is brought to the surface, the method of transferring the sample to the sample container is generally the same, with the exception that special requirements exist for VOC samples. The following procedures are based upon the method of subsurface soil sampling, and the most common method of transferring a grab sample to sample containers is discussed. For soil VOC sampling, very unique sampling methodologies must be employed which are described in Section 5. VOC samples shall not be homogenized or collected as composite samples.

4.4 Soil Sampling by Hand Auger Borers or Core Borers

Auger borings provide a simple method of soil investigation and sampling. Auger boring will be placed on a predetermined spacing grid with samples collected at predetermined depths, as outlined in the site Field Sampling Plan. The depth measurement will be marked on the shaft of the auger. A post-hole auger will be utilized for this procedure since this type of auger has a means of blocking the escape of soil from the auger (refer to ASTM D4547).

Make the auger boring by rotating and advancing the auger into the soil until the auger is full (generally just less than the length of the auger coring bit). Remove the auger and clean off and dump excess boring material when not at sampling depths. Before a sample is collected from a targeted sampling depth, remove all residual material from the auger and decontaminate the auger. To sample, carefully insert the auger into the borehole and rotate and advance until the auger is full. Collect the soil from the specified depth and place the soil in a stainless steel mixing bowl. Avoid collecting oversized rocks, roots, or other non-soil debris unless specified in the Field Sampling Plan. Collect enough soil volume from the desired depth to fill the required sample jar(s). Homogenize the soil in the mixing bowl, by the quartering method – divide the sample into quarters – mix each quarter thoroughly, and then remix all four quarters. From the homogenized soil, fill the sample jars as full as possible.

Obtain the required number of individual discrete samples from locations described in the Field Sampling Plan. A composite sample shall be comprised of not more than 8 discrete samples. For a composite sample over varying depth intervals, obtain discrete samples from determined intervals. In unstable soil a casing with an inside diameter slightly greater than the auger will be required. Advance the casing to a depth not greater than the top of the next intended sample depth, and clean the casing out with the auger. The auger can then be inserted into the borehole and rotated below the bottom of the casing to obtain the sample. This technique may be used when ground water is encountered, but consideration must be given to the loss of water while withdrawing the sample.

4.5 Soil Sampling by Split Spoon and Direct Push Methods

Advance the augers to just above the desired depth. Retract the auger and replace with a split spoon sampler. Record the number of blows to advance the split spoon. Alternatively, by Direct Push sampling, advance the stem with a sample barrel to the desired depth and obtain a soil sample. Collect enough soil volume from the desired depth to fill the required sample jar(s). For composite sampling, the individual discrete samples will be placed into a mixing bowl, and the material will be homogenized by using the method of quartering: divide the sample into quarters, mix each quarter thoroughly, and then remix all four quarters. From the homogenized composite soil, fill the sample jars as full as possible.

A composite sample shall be comprised of not more than 8 discrete samples. For a composite sample over varying depth intervals, obtain discrete samples from pre-determined intervals to be combined.

4.5.1 Composite Sample Collection

Composite samples are a means to gather data over a large area with minimal sample costs; however, lower resolution of contaminate delineation may result. Composite samples will not be obtained for VOC analysis. Composite samples are comprised from 2 but not more than 8 discrete samples. The individual discrete samples are collected in the same manner as grab samples. The individual discrete samples will be placed into a mixing bowl, and the material will be homogenized by using the method of quartering: divide the sample into quarters, mix each quarter thoroughly, and then remix all four quarters. From the homogenized composite soil, fill the sample jars as full as possible.

If the total sample volume is too large for a mixing bowl, then blending and sample size reduction will follow the standard procedures for "coning and quartering" described in ASTM C702.

5.0 SOIL VOC SAMPLES

Soil VOC samples must be obtained as grab samples in order that VOC volatilization is minimized. VOC samples shall not be homogenized or collected as composite samples. Excess disturbance or exposure to atmosphere of soils to be sampled for VOCs will cause any VOCs present to be volatilized, which will reduce the representativness of collected samples. Soil VOC samples should be collected as soon as possible after the soil has been exposed to the atmosphere and with as little disturbance of the soil as possible.

5.1 Soil VOC Sample Collection

The Field Sampling Plan will describe the method of Soil VOC collection. Many States are now requiring SW-846 Method 5035 which requires the use of hermetically sealed vials preserved with Sodium Bisulfate (for low anticipated VOC concentration) or Methanol (for high anticipated VOC concentration).

A small coring device EnCore ^{tm} or Purge and Trap Soil Sampler ^{tm} or a cut plastic syringe is used to obtain a soil plug (2-5 grams) from undisturbed soil. These devices are used to minimize soil disturbance and to trap VOCs in the soil during transfer to the soil vials. The soil face will be scraped clear of debris, smeared soil, rust from core-borers or split spoons, or other material that would interfere with obtaining a soil plug by the corer. The core will be obtained as quickly as possible (within a few minutes) from the soil after it has been brought to the surface from boring activities, slide hammer, or from a backhoe bucket.

Once the soil plug is obtained within the corer, the soil plug will be immediately transferred to the appropriate preserved soil vial, as described in the Field Sampling Plan or in the instructions supplied by the analytical laboratory. Gloves will be worn whenever handling the sample vials. The sample vials will be filled as completely as possible and wiped clean of dirt and moisture. The sample vials will be weighed to the nearest 0.01 grams. Alternatively, if prescribed in the Field Sampling Plan, trials to determine the weight versus length of soil column in the syringe will be performed, and the length of soil corresponding to 5.0 +/- 0.5 grams will used for future soil collections. The soil vials will never be opened in the field once soil has been deposited into the vials. Always handle the soil vials with gloves, before and after sample collection. More complete Method 5035 instructions can be obtained from the analytical laboratory or the manufacturer. Upon collection, VOC sample containers will be placed into plastic bags and placed upon ice in a cooler at 4 ^{o}C.

5.2 Headspace Analysis

Headspace analysis is a means of determining which portion of soil within a split spoon or core borer soil sample core has the highest VOC concentration. Headspace analysis is a crude field screening method and there are several variations of this field screening method. Variations include simply passing a PID or FID probe up and down the exposed soil sample core, while another method utilized for extruded sand from a Sonic Rig is to obtain a reading from the punctured plastic sample liner. The Headspace analysis method described below is a standard methodology.

If high humidity conditions exist during the time period when field activity is to be performed, the FID is recommended since a PID is not a reliable screening instrument under these conditions. The ionization potential of the lamp for the PID shall be optimal for the contaminants of concern.

Immediately upon opening the split-spoon (or other sample retrieval device) and after collecting the volatile organic sample (if required), a representative portion of the sample shall be collected and placed in a clean, contaminate-free jar. The sample may be placed in a new, clean, plastic sandwich bag inside a jar to minimize the number of new jars required. Additional headspace readings must be taken inside the plastic bag or sample jars prior to their use to ensure no external contamination is present.

If the volume of sample recovered is insufficient for all analytical requirements, then the material used in the headspace readings could be included with any nonvolatile sample matrix. Seal each jar with at least one continuous sheet of aluminum foil, using the jar lid to secure the foil. Vigorously agitate the sample jar for at least fifteen seconds - and then allow a minimum of ten minutes (or as the environmental conditions dictate) for the sample to adequately volatilize.

During cold weather, warm the samples to near room temperature prior to taking the headspace measurement. Re-agitate the jar and then remove the jar lid. Quickly insert the PID or FID probe through the aluminum foil and record the maximum instrument reading (which should be within the first two to five seconds). Erratic responses should be evaluated in terms of high organic vapor concentrations or conditions of elevated headspace moisture.

Record the headspace screening data on the boring log and any other appropriate documentation (e.g. sample transmittals, field notebooks, etc.) as appropriate.

Calibration of the screening instrument is performed with the appropriate calibration gas and shall be calibrated at least twice daily, and before use after a long shut down period (i.e. lunch breaks, equipment breakdowns, weather caused breaks, etc.).

If sample jars are to be re-used in the field, jars must be cleaned according to field decontamination procedures for the cleaning of sampling equipment.

PERMA-FIX ENVIRONMENTAL SERVICES			
TITLE:	Radiological Surveys	NO.:	RP-104
		PAGE:	1 of 9
		DATE:	May 2014

APPROVED:

5/31/14
Technical Services Manager Date

5/31/14
Corporate Certified Health Physicist Date

1.0 PURPOSE

This procedure establishes consistent methodology for performing radiation and contamination surveys at Perma-Fix Environmental Services (PESI) facilities and projects.

2.0 APPLICABILITY

This procedure is applicable to all personnel trained and qualified to perform radiation and contamination surveys at PESI.

3.0 REFERENCES

1. Title 17, California Code of Regulations, Division 1, Chapter 5, Subchapter 4 "Standards for Protection Against Radiation."
2. PESI "Radiation Protection Plan (RPP)
3. RP-101, "Access Control."
4. RP-105, "Unrestricted Release of Requirements."
5. RP-106, "Survey Documentation and Review

4.0 GENERAL

4.1 Discussion

Radiological surveys are performed to detect and assess radiological conditions, which may be encountered at PESI.

4.2 Definitions

Contact Dose Rate: A radiation dose rate as measured at contact or within 1/2 inch of the surface being measured.

CPM: Counts per minute

Dose Rate: The quantity of absorbed dose delivered per unit of time.

DPM: Disintegrations per minute

General Area Dose Rate (GA Dose Rate): The highest radiation dose rate accessible to any portion of the whole body measured at a distance of 30 cm (12 inches) from a significant radiation source or combination of sources.

LAW: Large area Wipe (i.e., Masslinn)

MDA: Minimum Detectable Activity

Survey: An evaluation of the radiation hazards incident to the production, use, release, disposal, or presence of radioactive materials or other sources of ionizing radiation under a specific set of conditions.

5.0 RESPONSIBILITIES

5.1 Radiation Safety Officer (RSO)

- Implementation of this procedure.
- Ensuring appropriate radiation surveys are performed to measure and document radiation levels.
- Ensuring all completed surveys are adequately reviewed.
- Providing technical direction to the RPTs.

5.2 Radiation Protection Technician (RPT)

- Conducting and documenting radiation surveys.
- Performing all necessary pre / post use operability checks.
- Creating neat, legible, and concise records.

6.0 PREREQUISITES

- Prior to performing a radiation survey, personnel should review previous survey data and familiarize themselves with possible radiological hazards.

7.0 PRECAUTIONS AND LIMITATIONS

- Personal Protective Equipment (PPE) should be appropriate for the level of contamination expected and shall be in compliance with Site Safety & Health Plan (SSHP), Radiation Work Permits (RWPs), or other work specific controlling documents. At a minimum, gloves or tweezers should be used when handling swipes.
- Direct probe surveys may be used to demonstrate compliance with removable limits given in Attachment 1 (Acceptable Surface Contamination Levels), and discussed in RPP-105, "Unrestricted Release of Requirements." When instrumentation is used in this manner it should be capable of achieving the removable minimum detectable count (MDC) requirements.
- Surface contamination limits are contained in Attachment 1.
- Instruments used in surveys should be capable of achieving a Minimum Detectable Activity (MDA) that is less than the applicable release limits.
- In high background areas it may not be possible to achieve the required survey MDAs for beta / gamma instruments.

8.0 APPARATUS

- Radiation and contamination survey instruments
- Smears
- Masslinn
- Personal Protection Equipment

9.0 RECORDS

Survey documentation to be completed per RPP-106, "Survey Documentation and Review."

10.0 PROCEDURE

10.1 General Instructions

1. Select the survey instrument based on the anticipated hazards and dose rates as determined by a review of previous survey data and ongoing work activities.
2. Perform pre-operational and response checks in accordance with the operating procedures for the instrument.
3. Remove any defective instrument from service.
4. Obtain survey forms and any other material required to document survey results.
5. Contamination Surveys are normally done for alpha emitting constituents. In certain circumstances the RSO can dictate that a survey be performed for both alpha and beta emitting constituents.

10.2 Routine Survey Frequencies

1. The RSO shall specify areas for routine monitoring surveys and the frequency of such surveys. The RSO should maintain a routine survey frequency schedule. The schedule is NOT considered a record, and does not need to be retained.
2. The following areas should be considered for a routine survey on a DAILY basis:
    - Access Control Points.
    - Designated eating, drinking, and smoking areas within Restricted Areas.
    - Radiological Counting Labs and sample prep areas.
    - Any other area specified by the RSO.
3. The following areas should be considered for a routine survey on a WEEKLY basis:
    - High Traffic areas on the PESI Site.
    - Operating high-efficiency particulate air (HEPA) exhaust areas.
    - Highly occupied areas within the radioactive Materials Area that could be a source of personnel contamination or an intake of radioactive materials (e.g., the boot change area, equipment floorboards, and workshops).
4. The following areas and equipment should be considered for a routine survey on a MONTHLY basis:

- Occupied offices.
- Storage areas.
- Occupied areas within the radioactive Materials Area that could be a source of personnel contamination or an intake of radioactive materials (e.g., equipment storage areas).

5. The following should be done on an as-needed basis:

   - Incoming Surveys

     The RSO can direct that incoming surveys be performed on equipment and materials arriving onto the site. The purpose of an incoming survey is to protect the client from financial liability associated with decontaminating equipment that arrived on the site with existing contamination. The degree of thoroughness of the survey and the requisite cleanliness of the equipment is at the discretion of the RSO.

   - Surveys of Materials Vehicles, and Personnel leaving Restricted Areas

     All materials, vehicles, and personnel shall perform surveys upon leaving Restricted Areas that have a potential for spread of contamination. The RSO or designee can direct that additional surveys be performed as needed to monitor for spread of contamination.

- Direct Total Contamination Surveys

  1. All items being surveyed should appear to be clean prior to being surveyed. To the extent possible, all interior and exterior surfaces should be free from oil and visible dirt. The RSO may dictate the required degree of cleanliness, based on the purpose of the survey and the history of the item being surveyed.

  2. Obtain proper instrumentation for the survey. Ensure that the instruments are currently calibrated and have been performance checked prior to the survey.

  3. Determine and record the background count in the area to be surveyed. Ensure that the background is representative of the measurement to be taken. Calculate and record the MDA on the appropriate survey form. Verify the MDA has been calculated for the background at the point of use and is less than the applicable site release criteria. In no case shall the background count time be less than the sample count time.

  4. Perform a scanning survey of the item. Concentrate survey measurements on areas most likely to be contaminated. The fraction of the total area scanned is subjective, based on technician experience, an item's use history, and RSO guidance. Typically, the scan frequency is a minimum of 10% of accessible surface areas.

  5. Obtain static measurements at locations with the highest potential for contamination. The number of survey points selected is subjective, based on technician experience, an item's use history, and RSO guidance. The count time should be consistent with the MDA calculation. A typical count

times is one minute for digital scalers and until the meter reading stabilizes for analog ratemeters.

6. Record and identify all locations surveyed on the appropriate survey form(s). The use of diagrams or sketches is recommended.
   - **Beta-Gamma Probe** - In high background areas it may not be possible to achieve the required survey MDAs. This should be noted on the survey cover sheet, and should be brought to the attention of the RSO.
   - **Alpha Probe** - The performance check background may be used in place of background count in the area to be surveyed. A good practice is to check the probe for light leaks or for faulty cables if positive results begin appearing.
7. All measurements shall be reported in units of "dpm" unless otherwise directed by the RSO. Examples include "dpm/100 cm^2," and "dpm/probe."
8. Direct non-smearable hot spots may be averaged over 1 square meter to determine compliance with release levels. If the entire item is less than 1 square meter in area, the entire surface area may be averaged. Bolt on parts of a vehicle should not be considered separate items.
   - The method for determining an average activity is to mark a 1 square meter area on the piece to be surveyed that is roughly centered on the hot spot. Take 1 measurement at the highest activity point of the hot spot. Take 4 (or more) other measurements within the square meter at locations representative of the whole square meter. Record count-rate of each individual measurement. Calculate the activity of all measurements being averaged, including those that are less than the MDA and those with a calculated activity less than zero. Calculate the average of all measurements and record on the survey form.
9. Complete the appropriate survey form.

10.3 Removable Contamination

With RSO approval, removable contamination surveys may be disregarded, provided that direct survey measurements and instrument MDAs are below site removable contamination limits for release.

1. All items being surveyed shall be clean prior to being surveyed. All interior and exterior surfaces should be free from oil and visible dirt. The RSO may dictate the required degree of cleanliness, based on the purpose of the survey and the history of the item being surveyed.
2. Wipe each location of interest with moderate pressure area using a standard 1 ¾-inch swipe. The area wiped should be approximately 100 cm^2. Larger areas may be wiped. It can be inferred that if the wipe meets the required limit for 100 cm^2 when it was actually taken from a larger area, the object will pass the 100 cm^2 criteria. No special documentation is required if the wiped area exceeds 100 cm^2. If the object is smaller than 100 cm^2, the area of the entire object should be wiped.

3. Large area wipes (LAW), also commonly referred to by the trade name "Masslinn" may be used to supplement smear surveys for removable contamination. The use of LAWs should be documented on the survey form with the notation "LAW," or equivalent.
4. Ensure each used swipe (i.e., smear or large area wipe) is handled, stored, and transferred in such a fashion as to prevent to loss of sampled material or cross-contamination with other personnel and other swipe samples.
5. Record the location of each wipe on the appropriate survey form. It is preferable to record the location by circling the sequential number location on a survey map where the wipe was taken.

10.4 Analyzing Swipes

1. Smear samples should be counted using available scintillation or gas-flow proportional laboratory counters, when practicable. Field instruments may be used for smear counting at the discretion of the RSO.
2. LAW samples may be counted using field instruments. The use of laboratory counters is inappropriate.
3. Determine and record the background count-rate. Calculate and record the MDA on the appropriate survey form. Verify the MDA has been calculated for the background at the point of use and is less than the applicable site release criteria. In no case shall the background count time be less than the sample count time.
4. Remove each swipe from the paper backing, as needed. The use of tweezers is recommended.
5. Place the swipe in the counter and close.
6. Count for the designated counting time.
7. Record the gross result under cpm in the appropriate column (either alpha or beta-gamma) of the survey form.
8. Calculate and record the activity. Removable contamination survey results shall be reported in units of "dpm" unless otherwise directed by the RSO. Examples include "dpm/100 cm^2" and "dpm/LAW."

10.5 Gamma Surveys

1. Routine gamma surveys may be used to detect the gradual buildup of gamma emitting contaminated materials in soils. This may occur at heavy equipment, heavy traffic, or egress points from contaminated areas. Normal uncontaminated trash should be gamma surveyed prior to leaving the site.
2. Obtain proper instrumentation for the survey. Ensure that the instruments are currently calibrated and have been performance checked prior to the survey.
3. Perform the survey with the appropriate detector using techniques specified by the RSO.
4. Complete the appropriate survey form.

10.6 Gamma Dose Rate Surveys

- Obtain proper instrumentation. Ensure that the instrument is currently calibrated and has been performance checked prior to the survey.
- When entering areas with known radiation levels, select the appropriate scale.
  - Observe the meters as you enter the area. If necessary, change scales to maintain on-scale reading.
- Perform gamma dose rate surveys as follows:
  - Monitor dose rates from the lower thighs to head level, recording the highest level as General Area Dose Rate.
  - Monitor dose rates 30 cm (12 inches) from a significant radiation source recording the highest level as General Area Dose Rate.
  - Additional measurements are necessary to determine Transport Index for shipping per procedure PP-8-810, "Conveyance Survey."
  - If dose rate sources are predominantly from overhead, then denote on survey.
  - Perform contact gamma dose rate measurements with the detector within ½-inch of the surface to be surveyed.
  - Additional measurement locations should be clearly identified in survey documentation.
  - Record all survey results on the appropriate survey form.

11.0 CALCULATIONS

11.1 Sample Activity

$$DPM = \frac{\left(\frac{TotalSampleCounts}{SampleCountTime}\right) - \left(\frac{TotalBkgCounts}{BkgCountTime}\right)}{(E)\,(A)}$$

where:

E = Instrument Efficiency
A = Area correction factor, if applicable

11.2 Minimum Detectable Activity (MDA)

The following MDA equation is to be used for a background count time equal to the sample count time:

$$MDA = \left(\frac{(3 + 4.65\sqrt{B})}{(E)(A)(T_S)}\right)$$

where:

Ts = Sample count time
E = Instrument efficiency
A = Area correction factor, if applicable
B = Background cpm

The following equation is to be used for a background count time equal to 5 or more times the sample count time:

$$MDA = \left(\frac{(3 + 3.29\sqrt{B})}{(E)(A)(T_S)} \right)$$

12.0 DOCUMENTATION

- Survey forms shall be completed in entirety. This includes attaching printouts, diagrams, or other supporting documentation, appending sequential page and survey tracking numbers, a review for completeness and accuracy, and appending the appropriate signatures of personnel performing the survey and / or analyzing samples.
- Once complete, the survey package shall be submitted to the RSO or designee, for final review and approval signature.
- Survey documentation shall be maintained according to established RP document control and retention requirements.

13.0 ATTACHMENT

Attachment 1 Acceptable Surface Contamination Levels

Attachment 1

Acceptable Surface Contamination Levels

NUCLIDE[a]	AVERAGE[b c] dpm/100 cm^2	MAXIMUM[b d] dpm/100 cm^2	REMOVABLE[b e] dpm/100 cm^2
U-nat, U-235, U-238 and associated decay products	5,000	15,000	1,000
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100	300	20
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000	3,000	200
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000	15,000	1,000

Notes:

[a] Where surface contamination by both alpha- and beta-gamma-emitting nuclides exists, the limits established for alpha- and beta-gamma-emitting nuclides should apply independently.

[b] As used in this table, dpm means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector for background, efficiency, and geometric factors associated with the instrumentation.

[c] Measurements of average contaminant should not be averaged over more than 1 square meter. For objects of less surface area, the average should be derived for each object.

[d] The maximum contaminated level applies to an area of not more than 100 cm^2.

[e] The amount of removable radioactive material per 100 cm^2 of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactive material on the wipe with an appropriate instrument of known efficiency. When removable contamination on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.

*Source: USCG / USEPA EM 385-1-80 Table 6-4 Acceptable Surface Contamination Levels, 1985.

Note: The acceptable surface contamination levels for Th-nat will be used unless subsequent sampling indicate the presence Ra-226, Ra-228, Th-230, Pa-231, or Ac-227 in concentrations greater than that of the parent nuclide. The RSO will determine if contamination limits should be modified for a specific activity or location based on available data.

PERMA-FIX ENVIRONMENTAL SERVICES			
TITLE:	Count Rate Instruments	NO.:	RP-108
		PAGE:	1 of 9
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APPROVED:

5/31/14
Technical Services Manager Date

5/31/14
Corporate Certified Health Physicist Date

1.0 PURPOSE

This procedure specifies the methods for set-up, daily pre-operational check, and operation of portable count-rate survey instruments. These instruments are used for the detection of radioactivity on personnel, on or within material surfaces, and in the environment. This procedure does not include associated instrument calibrations or cover the operation of exposure rate instruments.

2.0 APPLICABILITY

This procedure specifically addresses those meter-probe combinations that report values in units of counts or counts per minute (cpm) such as Ludlum Measurements models 2221 and 2241 Scaler-Ratemeters; and the Ludlum Model 177 Alarming Ratemeter or equivalent. These meters are mated to probes including the Ludlum Model 44-10, 44-20, and 44-62 NaI Detectors, the Ludlum Model 43-5 Alpha Scintillation Detector, and the Ludlum Model 44-9 Pancake Geiger-Mueller detectors or equivalent. Additional equivalent meters and probes may be used under this procedure without revision as approved by the RSO.

3.0 REFERENCES

1. ANSI N323A-1997, Radiation Protection Instrumentation Test and Calibration, Portable Survey Instruments.
2. Instrument Technical Manuals.
3. Perma-Fix Environmental Services (PESI) Radiation Protection Plan (RPP)
4. RP-104, Radiological Surveys

4.0 DEFINITIONS

cpm: counts per minute

DFSCL: Daily Field Source Check Logsheet.

dpm: disintegrations per minute

HV: High Voltage

MDA: Minimum Detectable Activity

5.0 RESPONSIBILITIES

5.1 Radiation Safety Officer (RSO)

- Reviewing and approving changes to this procedure and ensuring compliance with applicable regulations.
- Ensuring an adequate inventory of Radiation Protection instruments are available to support remediation activities.
- Overseeing the issue, control, and accountability of Radiation Protection instrumentation per the requirements of this procedure.
- Ensuring transmittal of all issue, control and accountability records to the appropriate document control authority when applicable.

5.2 Radiation Protection Technician (RPT)

- Maintaining instrument documentation and records as required by this procedure.
- Maintaining adequate instrument and equipment availability.
- Verifying current calibration and response test dates prior to issue or use of instruments.
- Promptly returning instruments to their proper location when work is complete.
- Ensuring that instruments are properly surveyed for contamination and decontaminated as necessary after use.

6.0 PREREQUISITES

- Only personnel with appropriate documented training shall issue or use RP instrumentation.
- Instruments and detectors shall be inspected for mechanical damage, and response tested prior to issue.
- Any instrument to be used shall have a current calibration label affixed to the instrument.

7.0 PRECAUTIONS AND LIMITATIONS

- Portable count rate survey instrumentations are susceptible to damage from physical and environmental stresses.
- QA/QC requirements established by an approved survey plan (e.g., Master Final Status Survey Plan) supersede the requirements of this procedure.

8.0 APPARATUS

- Appropriate survey instruments

9.0 RECORDS

- Portable Instrument Set-Up Sheet
- Daily Field Source Check Logsheet

10.0 PROCEDURE

10.1 General

1. Ensure the meter-probe combination selected is within their acceptable calibration periods. The swapping of probes between meters is permitted, but not encouraged. The following precautions and limitations must be observed and the following action steps must be taken:
    - If the meter-probe combination is calibrated as a set, Probe swapping is not permitted, without specific RSO approval.
    - The HIGH VOLTAGE (HV) and THRESHOLD settings for the meter-probe combination shall be identical. Note that the Ludlum 177 and 2241 do not have user adjustable settings for HV and THRESHOLD.
    - An initial set-up must be performed for each meter-probe combination prior to field use.
    - A source with known pedigree must be counted to verify the efficiency is within 10% of the calibrated efficiency, as applicable.
2. The RP Group will coordinate the calibration of boxes and probes on a minimum annual basis and after major repair operations. Battery and / or cable change-outs do not require re-calibration. Calibration procedures are outside of the scope of this instruction.
3. Pre-operational checks are required daily prior to use. Post-operational checks are performed as specified in work plans or procedures. Instruments used in the performance of daily activities do not normally require a post-operational check.
4. Instruments that fail operational checks or malfunction during use should be tagged or labeled "Out-of-Service" or "Do Not Use" and segregated from operational instruments. If possible, describe the problem on the tag / label and add initials and date.
5. Instruments leaving RP Group control (i.e., repair, calibration, excess, etc.) shall be surveyed for unconditional release according to the contamination criteria established in Table 1 of the Site RPP. The repair / calibration center may request a copy of the survey accompany any shipments of RP instruments.
6. Ensure meters with a "WINDOW" or "WIN" setting are set to "OUT."
7. Instruments may be operated in the FAST response mode if necessary. This setting is recommended if the audible response cannot be heard. SLOW response shall be used when performing instrument set-up and operational checks.
8. Ludlum NaI crystals are located in the end of the probe opposite of the cable connection. Use this end for surveys.
9. Calibration stickers are attached to the instruments and detectors. Illegible stickers should be replaced prior to instrument use.
10. Instrument set-up and subsequent operational checks should be performed in the same location, with consistent temperature and background radiation levels.

11. Source positioning devices (i.e., jigs) may be used to ensure a reproducible geometry between instrument checks. Source geometry must be consistent between initial instrument set-up and subsequent operational checks.

12. Instruments that do not have scaler capability should be set-up and checked by replacing 1-minute timed counts with static count rate measurements. Each static measurement should last until the meter reading fully stabilizes.

10.2 Instrument Set-Up

1. Inspect the meter-probe combination for physical damage or defect.

2. Complete Section A of the Portable Instrument Set-Up Sheet (Attachment 1).

3. Perform 10 1-minute source counts alternating with 10 1-minute background counts. Remove / replace the source and reposition the probe after each count. During alternating background counts, ensure that the source is sufficiently shielded so as not to impact background values.

   NOTE: Counts (Source and Background) performed with a Ludlum 43-5, or other large surface area probe, should be alternated between the Heel, Center, and Toe Positions, if the source surface is smaller than the active surface area of the probe. Instrument response can vary greatly across the probe surface.

4. Document each count on the Portable Instrument Set-Up Sheet.

5. Calculate and record the net count value by subtracting the corresponding background count from each source count.

   NOTE: Determining Sigma (Standard Deviation) values is useful when specific plans or activities require higher data quality objectives and / or when the development of control charts is necessary.

6. Calculate and record the following values from the obtained background counts:

   - Avg. Value (Sum of values / # of counts)
   - Sigma Value (Standard Deviation of all counts)
   - 20% Value (Avg. Value * 0.20)

7. Calculate and record the +/- 20% Values and the +/- 1, 2, and 3 Sigma values using the AVG. VALUE as a reference point.

8. Repeat the previous two steps for determining NET COUNT acceptable ranges. The 3 Sigma value must be less than the +/- 20% value.

9. Obtain a blank Daily Field Source Check Logsheet (DFSCL) (Attachment 2) and transfer the instrument, source, and acceptable range data, as applicable, from the Portable Instrument Set-Up Sheet.

10. Place the DFSCL in the designated use location and forward the completed Portable Instrument Set-Up Sheet and submit to the RSO, or designee for review.

11. Ensure sources are stored properly after use in the designated source storage location.

10.3 Operational Check

1. Obtain the selected meter-probe combination and corresponding DFSCL (Attachment 2).
2. Record the date and time on the DFSCL.
3. Perform and document the following checks on the DFSCL, as applicable:
    - Perform a physical inspection. Observe for instrument damage. Alpha probes should be checked for light leaks by inverting the probe face towards a light source and observing instrument response. If the instrument fails to respond at all or over-responds this may be an indication of a light leak and should be investigated further, prior to proceeding.
    - Perform a battery check. Instrument Models differ in method. Some meters have a visible battery range on the meter face. The Ludlum Model 2241 has a battery indicator in the digital display that lights if the batteries require replacement. The Ludlum Model 2221 has a BAT button that brings up the battery level in the digital display. Ensure this value is at least 5.0v. Change batteries and retest as necessary.
    - Verify and adjust the HV, when possible, to match the initial set-up data. Minute differences in HV (+/- 5v) are acceptable without adjustment.
    - Perform an audio response check.
4. Perform and record a 1-minute background count. Report any abnormal background responses to the RSO, prior to instrument use. Normally acceptable background levels < 5 cpm for alpha probes, and < 300 cpm for Pancake G-M probes. Acceptable background levels for NaI probes are variable due to crystal size and based on technician experience.
5. Perform and record a 1-minute source gross count using the same source and geometry applied during initial set-up.
6. Calculate and record the net count value.
7. Compare the net count value to the acceptable range. If the instrument response is outside the acceptable range, the process may be repeated a maximum of 1 additional time before placing the instrument out-of-service.
8. If the instrument fails the pre-operational checks, mark FAIL, initial the DFSCL, and place the instrument out-of-service. Deliver completed DFSCL to the RSO or designee, and explain the failed condition(s).
9. If all checks pass, mark PASS, initial the DFSCL, and return form to designated in-use storage location. This may be a binder, folder, or cabinet. The instrument is now ready for use.
10. If the instrument will be used for routine personnel exit monitoring ensure the alarm threshold is set to alarm and actuates at a level below the site removable contamination limits identified in Table 6-1 of the Site Safety & Health Plan (SSHP). Make adjustments as necessary.

11. Ensure sources are stored properly after use in the designated source storage location.

10.4 Operations

1. Operate instrument in a manner that minimizes the potential for cross-contamination and physical damage.
2. Evaluate the surface or area to be surveyed for potential scanning interferences. For example, thin layers of water or soil can prevent the detection of alpha contamination. Another example is the use of a NaI probe to qualify soil contamination. The presence of standing water can have a significant impact on instrument response. Initiate necessary corrective actions prior to survey or note conditions during survey reporting.
3. Most instruments will operate in temperatures between 10 and 120 degrees Fahrenheit. However, anytime the temperature is outside of the 32 degree (freezing) or 100 degrees ranges, observe the following precautions:
    - Use particular caution with NaI crystals that may shatter under extreme temperature changes. If the temperature difference is greater than 30 degrees between storage and usage locations, wrap the probe tightly in a cloth towel or other insulator and allow warming or cooling over at least one hour prior to use.
    - Periodically check the instrument against a known source of radiation or contamination. If the instrument appears to be responding incorrectly contact the RSO or designee for guidance.
    - Contact the RSO for guidance anytime work is planned outside of the 10 to 120 degree range.
4. Protect instruments to the extent possible from exposure to moisture (i.e., rain, snow, etc.) during use. Instruments shall be stored in a safe manner when not in use.
5. Minimum Detectable Activities (MDA) for each survey should be determined by evaluating field background levels, not background values obtained during operational checks. Calculate MDA using the formula provided in PP-8-805, "Radiological Surveys."
6. Determining activity in disintegrations per minute (dpm) should be performed using the instrument efficiency obtained during calibration. Efficiencies are normally not established for NaI probes, and therefore should not be used for quantifying activity concentrations. The use of NaI probes for activity quantification shall be evaluated by the RSO prior to performance.
7. Observe the following when performing survey scans and static measurements:
    - Alpha probes should be held within ¼-inch of the surface being surveyed. Probe speed should not exceed 1 probe width per second.
    - Beta probes should be held within ½-inch of the surface being surveyed. Survey speed should not exceed one probe width per second.

- NaI probes should be held as close as possible to the surface being surveyed without contaminating the probe housing. Note that the crystal is located in the probe end opposite the cable connection. Use appropriate sleeving or wrapping in wet or dirty environments.
- The scan speed for performing Gamma Walkover Surveys is approximately 0.5 m/sec. Move the detector side to side using a 1-meter path length. Each side-side swing should take 2 seconds to traverse the 1-meter path. Advance the probe forward as you go at a rate of approximately 0.5 m/sec. Use the audio function. When increased counts are detected, slow down and locate the source as would be done in a normal survey. Walk parallel paths to ensure that 100% of the area is surveyed. Ensure that the survey extends to the boundaries of the survey unit. Pay particular attention to low lying areas, ditches, and points of possible contamination.
- Static measurements should be performed in any location were scans indicated the presence of activity. This is due to the fact that instrument MDAs are normally based on a 1-minute static measurement.
- All static measurements should be at least 1 minute, if the instrument has a scaler function. If the instrument is a ratemeter only, static measurements should last until the meter reading has fully stabilized.

8. Perform a post-operational check after use if directed by work plan, procedure, or the RSO.

11.0 ATTACHMENTS

Note: Attachments may be revised without formal review of this procedure and are attached as examples only. Please contact the RSO for a current copy of these attachments.

Attachment 1 Portable Instrument Set-Up Sheet (Typical)

Attachment 2 Daily Field Source Check Logsheet (Typical)

Attachment 1
Portable Instrument Set-Up Sheet (Typical)

PORTABLE INSTRUMENT SET-UP SHEET

Set-Up Location: _____

INSTRUMENT DATA

	INSTRUMENT	DETECTOR
MODEL		
SERIAL #		
CAL DUE		
HV		
THRESHOLD		

SOURCE DATA

ISOTOPE	
SERIAL #	
ACTIVITY (uCi)	
ACTIVITY (dpm)	

COUNT (n)	Source Counts	Source Count Time (min)	Source CPM	Background Counts	Background Count Time (min)	Background CPM	NET CPM
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

CALCULATED VALUES

Background (CPM)	Net CPM
Average	
+/- Sigma	
+/- 20 %	

ACCEPTABLE RANGES

Background (CPM)	Net CPM
+ 20 %	
+ 3 Sigma	
+ 2 Sigma	
+ 1 Sigma	
- 1 Sigma	
- 2 Sigma	
- 3 Sigma	
- 20 %	

REMARKS

Performed By: Date / Time:

Reviewed By: Date / Time:

Attachment 2
Daily Field Source Check Logsheet (Typical)

DAILY FIELD SOURCE CHECK LOG

MONTH / YEAR: _____

INSTRUMENT DATA			Date/Time	Physical	Battery	High Voltage	Audio	Background CPM (A)	Source CPM (B)	Net CPM (C)	PASS or FAIL	Tech. Initials
	INSTRUMENT	DETECTOR										
MODEL												
SERIAL #												
CAL DUE												
SOURCE DATA												
ISOTOPE												
SERIAL #												
ACTIVITY dpm												
INSTRUMENT RANGES												
	Background	Net CPM										
+ 20 %												
+ 3 Sigma												
+ 2 Sigma												
+ 1 Sigma												
- 1 Sigma												
- 2 Sigma												
- 3 Sigma												
- 20 %												
NET CPM CALCULATION (B) - (A) = (C)												

Remarks:

Reviewed by:

PERMA-FIX ENVIRONMENTAL SERVICES			
TITLE:	Dose Rate Instruments	NO.:	RP-109
		PAGE:	1 of 8
		DATE:	May 2014

APPROVED:

5/31/14

Technical Services Manager Date

5/31/14

Corporate Certified Health Physicist Date

1.0 PURPOSE

This procedure specifies the methods for performing source checks and operating portable Gamma scintillation dose rate instruments, specifically, the Ludlum Model 12s uR and the Bicron Model Micro Rem. These instruments are used for the evaluation of exposure rates from radioactive materials and determining environmental radiation levels.

2.0 APPLICABILITY

This procedure addresses those instruments that measure dose rate from a scintillation detector and have displays that read in uR/hr, uRem/hr and/or mRem/hr such as Ludlum 12s, Bicron Micro Rem, or Eberline RO-2. Equivalent instruments that operate in a similar fashion to those identified in this section may be operated under this Project Procedure with RSO approval.

3.0 REFERENCES

1. ANSI N323-1978, Radiation Protection Instrument Test and Calibration.
2. Instrument Technical Manuals.
3. Perma-Fix Environmental Services (PESI) RPP

4.0 DEFINITIONS

None

5.0 RESPONSIBILITIES

5.1 Radiation Safety Officer (RSO)

- Reviewing and approving changes to this procedure and ensuring compliance with applicable regulations.
- Ensuring an adequate inventory of Radiation Protection instruments are available to support remediation activities.
- Overseeing the issue, control and accountability of Radiation Protection instrumentation per the requirements of this procedure.

- Ensuring transmittal of all issue, control and accountability records to the appropriate document control authority when applicable.

5.2 Radiation Protection Technician (RPT)

- Maintaining instrument documentation and records as required by this procedure.
- Maintaining adequate instrument and equipment availability.
- Verifying current calibration and response test dates prior to issue or use of instruments.
- Promptly returning instruments to their proper location when work is complete.
- Ensuring that instruments are properly surveyed for contamination and decontaminated as necessary, after use.

6.0 PREREQUISITES

- Only personnel with documented training shall issue or use RP instrumentation.
- Instruments and detectors shall be inspected for mechanical damage, and response tested prior to issue.
- Any instrument to be used shall have a current calibration label affixed to the instrument.

7.0 PRECAUTIONS AND LIMITATIONS

- Portable count rate survey instrumentations are susceptible to damage from physical and environmental stresses.

8.0 APPARATUS

- Survey instrument
- Tech source
- Source positioning device (jig)

9.0 RECORDS

- Daily Field Source Check Log – Exposure Rate Instruments (Attachment 1)
- Exposure Rate Instrument Set-Up Sheet (Attachment 2)

10.0 PROCEDURE

10.1 General

1. Ensure the instrument selected is within their acceptable calibration periods. This is indicated on an attached calibration sticker. Illegible stickers should be replace prior to instrument use.
2. The RP Group will coordinate instrument calibration on a minimum annual basis and after major repair operations. Battery change-outs do not require re-calibration. Calibration procedures are outside of the scope of this instruction.
3. Pre-operational source checks are required daily, or prior to each intermittent use, whichever is less frequent. Post-operational source checks are performed

as specified in work plans or procedures. Instruments used in the performance of daily activities do not normally require a post-operational source check.

4. Instrument set-up and subsequent operational checks should be performed in the same location, with consistent temperature and radiation background levels.
5. Use a gamma check source with an activity sufficient to produce contact exposure rates at least ten times higher than background. Cs-137 is typically since it emits 662 keV gamma rays which are representative of the mid-range of gamma energies encountered at NFSS. Alternate sources may be used with RSO approval.
6. Source positioning devices (i.e., jigs) should be used to ensure a reproducible geometry between instrument checks. Source geometry must be consistent between initial instrument set-up and subsequent operational checks.
7. The Ludlum 12s may be operated in the FAST response mode. Switch to SLOW response for obtaining precise readings.
8. Internal scintillation crystals are orientated towards the front of the instrument. Meter cases have visible indicators showing optimum locations to obtain measurements (i.e. effective detector center).
9. Allow instrument readings to maximize prior to recording instrument reading. This may take up to twenty seconds. Note that the needle may not rest on a single value, but may fluctuate slightly between two points on the scale. If this is the case, an average reading should be obtained by summing these two end points and dividing by two.
10. Instruments should be allowed to warm-up for at least one minute prior to obtaining readings.
11. Report any abnormal instrument readings (e.g., unstable analog meter fluctuations), or background inconsistencies to the RSO, prior to continuing instrument use.
12. Instruments that fail operational checks or malfunction during use should be tagged or labeled "Out-of-Service," or "Do Not Use," and segregated from operational instruments. If possible, describe the problem on the tag / label and add initials and date.
13. Instruments leaving RPP Group control (i.e., repair, calibration, excess, etc.) shall be surveyed for unconditional release. The repair / calibration center may request a copy of the survey to accompany shipments of RP instruments.

10.2 Instrument Source Check

1. Obtain the selected instrument.
2. Obtain the corresponding Daily Field Source Check Log – Exposure Rate Instruments form, Attachment 1. This form will be referred to as the "Source Check Log." Initiate a new Source Check Log, if necessary.
3. Perform a physical inspection of the instrument. Place particular emphasis on the following items:

- Instrument case is not visibly damaged beyond minor scrapes and scratches.
- Analog display is not cracked or otherwise damaged.
- Switches and buttons are functional.
- Audio, if present, is functional.
- Calibration labels are legible and instrument is within calibration period.

4. Note results of physical inspection on the Source Check Log.
5. Verify the battery level is within the acceptable range on the analog display. Replace batteries and re-verify, as necessary.
6. Note battery check results on the Source Check Log.
7. Verify the high voltage (HV) level is within the acceptable range on the analog display, if present. Place the instrument out-of-service if the HV is outside the acceptable range.
8. Note the HV check results on the Source Check Log.
9. If acceptable background ranges have not been established, perform the following:
    - Obtain a blank NFSS Exposure Rate Instrument Set-Up Sheet, Attachment 2. This form will be referred to as the "Set-Up Sheet."
    - Record the basic source and instrument information at the top of the form.
    - Using the instrument and the source jig (without source), obtain and record ten background readings. The instrument should be removed from the source jig and repositioned after each reading is obtained. Make sure the location where readings are obtained has stable background levels and is the location used for subsequent source checks.
    - Calculate and record the average background value and +/- 20% values on both the set-up and source check logsheets.
10. Obtain and record an average background reading on the source check log.
11. Compare the average background reading to the acceptable range. If background response is outside this range, report the condition to the RSO for evaluation, otherwise continue with source check process.
12. Obtain the source to be used for instrument source checks.
13. If acceptable source check ranges have not been established, perform the following:
    - Obtain the Set-Up Sheet used to determine acceptable background ranges for the instrument.

- Using the instrument and the source jig (with source), obtain and record ten contact source readings. The instrument and source should be removed from the source jig and repositioned after each reading is obtained. Make sure the location where readings are obtained is the same location where previous background readings were obtained.
- Calculate and record the average source value and +/- 20% values on both the set-up and source check logsheets.

14. Load the source and instrument onto the source jig.
15. Obtain and record the "CONTACT" reading.
16. Verify the contact reading is within the acceptable range (+/- 20%).
17. If the contact source reading falls outside the acceptable range, tag the instrument out of service and notify the RSO, otherwise continue.
18. Complete the source check log including technician initials. The instrument is now ready for use.
19. Ensure sources and forms are stored properly after use in the designated storage location. Forms are retained in RP Instrument logbooks of field files during instrument use (i.e. calibration) cycle. Records are then reviewed by the RSO, or designee for completeness and forward to Project Records for retention.

10.3 Operations

1. Verify that required source checks have been performed prior to initial instrument use.
2. Operate instrument in a manner that minimizes the potential for cross-contamination and physical damage.
3. Limit readings taken while the instrument is positioned sideways to minimize the effects of "geotropism" on the analog needle.
4. Obtain readings by positioning the instrument as close to the detector's "effective center" as possible. The detector effective center is represented on the instrument housing a cross inside a circle on the Bicron Micro Rem, and a small circular depression on the Ludlum 12s. Overall optimum readings are collected from the front of the instrument housing.
5. Most instruments will operate in temperatures between 10 and 120 degrees Fahrenheit. However, anytime the temperature is outside of the 32 degree (freezing) or 100 degree ranges, observe the following precautions:
    - Be observant of instrument response to background. If the instrument begins to show a decreased response to expected background levels contact the RSO, or designee for guidance.
    - If practicable, perform a period response check of the instrument against a known source of radiation. If the instrument appears to be responding incorrectly contact the RSO or designee for guidance.
    - Contact the RSO for guidance anytime work is planned outside of the 10 to 120 degree range.

6. Protect instruments, to the extent possible, from exposure to moisture (i.e. rain, snow, etc.) during use. Instruments shall be stored in a safe manner when not in use.
7. Perform a post-operational source check after use, if directed by work plan, procedure, or the RSO.

11.0 ATTACHMENTS

Attached forms are examples and may be modified by the RSO, as needed, without revision to this procedure.

Attachment 1 Daily Field Source Check Log – Exposure Rate Instruments (Typical)

Attachment 2 Exposure Rate Instrument Set-Up Sheet (Typical)

Attachment 1

Daily Field Source Check Log – Exposure Rate Instruments (Typical)

FMSS DAILY FIELD SOURCE CHECK LOG
- EXPOSURE RATE INSTRUMENTS

MONTH / YEAR: _____

INSTRUMENT DATA	Date/Time	Physical	Battery	High Voltage	Audio	Background	Contact Source	PASS or FAIL	Tech. Initials
INSTRUMENT									
MODEL									
SERIAL#									
CAL DUE									
HV									
SOURCE DATA									
ISOTOPE									
SERIAL #									
ACTIVITY uCi									
INSTRUMENT RANGES									
	Background	Contact Source							
+ 20 %									
- 20 %									
Units (Circle One									
uR urem mR mrem R rem									
Remarks:									

Reviewed by: _____

Attachment 2

Exposure Rate Instrument Set-Up Sheet (Typical)

FMSS EXPOSURE RATE INSTRUMENT SET-UP SHEET

Set-Up Location: _____

INSTRUMENT DATA		READING (n)	Background Rate	Contact Source Rate	CALCULATED AVERAGE AND RANGES	
	INSTRUMENT				Background	Contact Source
MODEL		1				
SERIAL #		2			Average + 20%	
CAL DUE DATE		3			Average	
HV		4				
		5			Average - 20%	
SOURCE DATA		6			Units (Circle One)	
ISOTOPE		7			uR urem mR mrem R rem	
SERIAL #		8				
ACTIVITY (uCi)		9			REMARKS	
		10				
Performed By:		Date/Time:	Reviewed By:		Date/Time:	

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Title: Gasoline Range Organics (GRO) by GC/FID [SW846 Method 8015 and State Methods]

Approvals (Signature/Date):

Gabe Ornelas — 6/3/20 — Date Technical Specialist	Reed Pottruff — 6/3/20 — Date Health & Safety Manager / Coordinator
Roxanne Sullivan — 6/4/20 — Date Quality Assurance Manager	Scott Hall — 6/4/20 — Date Laboratory Director

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1.0 Scope and Application

GRO components correspond to an alkane range from n-hexane (C_6) to n-decane (C_{10}), with boiling points between approximately 60 ℃ and 170 ℃. The method is sensitive to a variety of aromatic and aliphatic hydrocarbons that can be present in a wide range of petroleum distillates and petroleum products (e.g., automotive gasoline, jet fuels, lighter fluid, and some paint strippers). It may be necessary to modify the scope to include other ranges as specifically requested by a client driven QAS.

1.1 Analytes, Matrices, and Reporting Limits

1.1.1 This method is used to determine the concentration of gasoline range organic (GRO) materials in water and soil. This procedure is based on EPA Method 8015B, 8015C, 8015D, and associated methods 8000B and 8000C. Also represented are Alaska Method AK101, Washington method NWTPH-Gx **and** Oklahoma Methods 8020/8015 (modified Gasoline Range Organics). The standard laboratory reporting limits (RLs) for these methods are listed in the following table:

Method	Lab Reporting Limit - Water (mg/L)	Method Reporting Limit - Water (mg/L)	Lab Reporting Limit - Soil (mg/kg)	Method Reporting Limit - Soil (mg/kg)
8015	0.025	--	1.2	--
NWTPH-Gx	0.025	0.250	1.2	5
AK101	0.025	0.100	1.2	20
Oklahoma	0.025*	0.020	0.5*	0.1

* The laboratory MDL does not support the Oklahoma method's lower RLs

1.1.2 Unless otherwise noted the most stringent or common QC criteria are reported and adhered to for the methods listed. Consistent with SW-846, method references will not reference a specific version unless it is necessary to state criteria for that version.

2.0 Summary of Method

2.1 Water samples are analyzed directly for gasoline range organics using the SW-846 Method 5030 purge-and-trap procedure and Method 8015 GC analysis. An inert gas is bubbled through a portion of the aqueous sample at ambient temperature, and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are adsorbed. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto the gas chromatograph column for analysis.

2.2 Solid samples are preserved in and extracted with methanol by the SW-846 Method 5035 high-level option. An aliquot of the methanol extract is added to organic-free reagent water, which is then analyzed using the SW-846 Method 5030 purge-and-trap procedure and Method 8015 GC analysis. The Alaska AK101 method requires

dispersion of soil and waste samples in methanol that contains the method surrogates at the time of sampling in the field to dissolve and preserve the volatile organic constituents.

2.3 Method 8015 is a gas chromatography (GC) method. Detection is achieved by a flame ionization detector (FID), which can be used with a photoionization detector (PID) in series to characterize individual aromatic components by method 8021B (see SOP DV-GC-0023 for details). The GRO retention time range is characterized by running the specified marker compounds as outlined in the definitions section below. Quantitation of GRO organics is based on the FID detector response to an external gasoline standard.

3.0 Definitions

3.1 Gasoline Range Organics (Gasoline): All chromatographic peaks eluting between the peak start of 2-Methylpentane and the peak end time for 1,2,4-Trimethylbenzene are attributed to GRO. Quantitation is based on a direct comparison of the area within this range to the total area of the calibration standard within this range.

3.2 C6-C10: All chromatographic peaks eluting between the peak start of 2-Methylpentane and the peak end time for Dodecane are attributed to this range. Quantitation is based on a direct comparison of the area within this range to the total area of the calibration standard within this range.

3.3 C6-C12: All chromatographic peaks eluting between the peak start of 2-Methylpentane and the peak end time for Dodecane are attributed to this range. Quantitation is based on a direct comparison of the area within this range to the total area of the calibration standard within this range.

3.4 Alaska Method AK101 defines GRO as all chromatographic peaks, both resolved and unresolved, eluting between the peak start time for C_6 (n-hexane) and the peak start time for C_{10} (n-decane). Quantitation is based on a direct comparison of the baseline-to-baseline integrated area within this range to the total area of the calibration standard over the same ($C_6 – C_{10}$) range, using FID response.

3.5 Oklahoma Methods 8020/8015 (modified) defines GRO as all chromatographic peaks, both resolved and unresolved, eluting 0.1 minutes before the retention time for MTBE and 0.1 minutes after the retention time for naphthalene. Quantitation is based on a direct comparison of the area within the range to the area of a ten component blend of typical gasoline compounds as specified in Table 3.

3.6 Method NWTPH-Gx requires the retention time range (window) for gasoline integration to include, at a minimum, toluene through naphthalene. The standard NWTPH_Gx range are all chromatographic peaks, both resolved and unresolved, eluting between the peak start time for 2-methylpentane and the peak end of C_{12} (dodecane), using a baseline-to-baseline integration.

3.6.1 Surrogate peak areas must be determined by valley-to-valley integration. Surrogate peak areas get subtracted out of the total GRO area by the software.

3.6.2 Other volatile petroleum products may be analyzed by method NWTPH-Gx provided that they elute within the gasoline range and the window is adjusted to

encompass the expected range of the product and co-eluting surrogate areas are subtracted.

3.7 User defined ranges (8015 modified), unless otherwise specified and agreed upon by the client, defines GRO as all chromatographic peaks, both resolved and unresolved, eluting between the peak start time for earliest representative alkane marker and after the peak start time for latest representative alkane marker. In the case of the range C_4-C_{12} the range includes all chromatographic peaks, both resolved and unresolved, eluting after the methanol solvent peak and before the peak start time of the tridecane peak. See section 10.5 for guidance about how to incorporate the RT window into the specified hydrocarbon range.

Note: Because n-butane is a gas at room temperature, the laboratory cannot use this compound to establish the beginning of the retention time range for C_4. As a result, TestAmerica Denver does not have any data that includes the earliest C_4 compounds encompassed in the C_4 range. It is likely that some of the early compounds would be unresolved from the methanol peak or possibly not retained on the trap. The integration is started as soon after the methanol solvent peak as feasible to include any peaks resolved from the methanol solvent peak.

3.6 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, "Quality Assurance Program," for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 High levels of heavier petroleum products such as diesel fuel may contain some volatile components producing a response within the retention time range for GRO. Other organic compounds, including chlorinated solvents, ketones, and ethers, are also detectable by this method. As defined in the method, the GRO results will include these compounds. In cases where an interfering compound co-elutes with a surrogate the sample results will not include that response in the GRO calculation and the results may be biased low. An NCM will be written to address this issue when it occurs.

4.2 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank prepared from reagent water (for water samples) or methanol (for soil and sediment samples that will be preserved with methanol in the field) and carried through sampling and subsequent storage and handling can serve as a check on such contamination. A method blank is prepared with each batch in order to monitor interferences that may be present in the solvents, glassware, and reagents. Reagent water is prepared from the zero grade water system by boiling the water while bubbling nitrogen gas through the water. The water is transferred to a dispenser while still very warm.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of an instrument blank to check for cross contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bake-out and purging of the entire system may be required. Screening of all samples prior to analysis is recommended to protect analytical instrumentation.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

5.2.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

5.2.2 A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Gas chromatographic analytical system complete with gas chromatograph suitable for purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system capable of determining peak areas is recommended.

6.1.2 The instruments used are:

6.1.2.1 Instrument Q- Agilent 6890A with an OI 4552 autosampler and a Tekmar 3000 purge and trap unit.

6.1.2.2 Instrument S1- Agilent 6890N with an EST Centurion autosampler and an Evolution purge and trap unit.

NOTE: Equivalent instruments may be used.

6.1.3 Columns

6.1.3.1 Instruments Q and S1 use a Restek 502.2 that is 60 m with a 0.53 mm I.D

6.1.3.2 Other capillary columns such as a 30 m, 0.53 mm ID, DB-5 may be used, but the column must be able to resolve 2-methylpentane from the methanol solvent front in a 30 ∝g/L LCS standard.

6.1.4 Detectors: Flame ionization detector (FID) is used for the measurement of all hydrocarbons.

6.1.5 Purge-and-Trap Devices: EST Evolution with a J or M trap or equivalent and a Tekmar 3000 or equivalent .

6.1.6 Balances - The accuracy of each balance is checked each day of use (see SOP DV-QA-0014 for details).

6.1.6.1 An analytical balance capable of accurately weighing 0.0001 g is used for preparing standards.

6.1.6.2 A top-loading balance capable of weighing to the nearest 0.01 g is used for weighing samples. .

6.1.6.3 The NWTPH_Gx method requires the use of a balance capable of weighing to the nearest 0.001 g.

6.2 Supplies

6.2.1 Syringes: 5 mL glass.

6.2.2 Class A Volumetric Flasks: 10 mL, 50 mL, 100 mL, 500 mL, and 1,000 mL with a ground-glass stopper.

6.2.3 Microsyringes: 1 µL, 5 µL, 10 µL, 25 µL, 50 µL, 100 µL, 250 µL, 500 µL, 1000 µL and 2500 µL.

6.2.4 Glass Scintillation Vials: 20 mL, with screw-caps/crimp caps and Teflon liners or glass culture tubes, or equivalent.

6.2.5 Stainless steel spatula.

6.2.6 Wooden tongue depressors.

6.2.7 Disposable Pasteur pipettes

6.2.8 pH paper: Wide range.

6.2.9 Vials: 2 mL with screw caps and Teflon liners.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on G:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current version for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Reagent Water: De-ionized reagent water that is boiled and purged with nitrogen.

7.1.2 Methanol: Purge and trap grade or equivalent. Store away from other solvents. See SOP CA-Q-S-001 for the description of the program for testing solvents prior to use.

7.1.3 Methanol: HPLC grade, used for cleaning labware.

7.1.4 Sand: Reagent grade Ottawa Sand or equivalent, dried in an oven at 105 ℃ for 8 hours before using.

7.2 Gasoline Calibration Standards

NOTE: Specifics for preparing diluted standard solutions depend on the concentration of the original stock standard. The source of the stock or parent neat gasoline standard depends on the specific client or project requirements and can change from project to project. The details presented in this section are intended to provide guidance only. Specific dilutions may change depending on the source of the gasoline standard required for the project.

7.2.1 Gasoline Standards Sources

Authentic gasoline fuel standards are used for calibrations and spiking. Depending on project/client requirements, the gasoline standard may be any of the following:

7.2.1.1 For 8015 GRO methods, Alaska Method AK101 and NWTPH-Gx a regular unleaded gasoline primary source standard (**8015GROCSAStk**) at 10,000ug/mL will be used, purchased as a certified mix of gasoline and aromatic hydrocarbons (BTEX). A neat (100%) gasoline obtained from a gasoline station, either locally or from the sampling region, as directed by client requirements can also be used. See Table 1.

7.2.1.2 For Oklahoma GRO a ten component blend of typical gasoline compounds obtained as a concentrated stock standard solution in methanol from a commercial source at 10,000ug/mL (**OKGRO CSA Stk**) will be used a primary source. This standard serves as the stock quantitation standard.See Table 3.

7.2.2 Preparing GRO Standards from Neat Gasoline

7.2.2.1 To prepare a stock standard from neat gasoline, first determine the density of the neat standard by weighing 2 ∝L of the standard in a tared 10 ∝L syringe. The density is calculated as follows:

$$\rho = \frac{m}{v}$$ *Equation 1*

Where:

ρ	=	Density in g/∝L
m	=	Mass in grams
v	=	Volume in ∝L

7.2.2.2 Preparation of GRO Stock Standard, 10,025 ∝g/mL

To prepare a stock standard from neat gasoline, dilute a volume of the neat gasoline, as determined based on its density, to 5 mL with methanol to achieve a final concentration of 10,025 ∝g/mL. The volume of the neat gasoline needed is determined as follows:

$$\text{Volume of Neat Gasoline } (\propto L) = \frac{10025(\propto g / mL) \times 5(mL)}{\rho(g / \propto L) \times 1000(\propto g / g)}$$ *Equation 2*

This calculation will generally yield a result somewhat greater than 50 ∝L.

7.2.3 Commercial Calibration Stock Standard, 10,025 ∝g/mL

As described in Section 7.2.1.1, a GRO stock standard may be prepared using a neat gasoline standard. Alternatively, a stock GRO or combined GRO and aromatic solvent (BTEX) stock standard may be purchased from a commercial vendor with a GRO concentration of 10,000 ∝g/mL in methanol. See Tables 1 - 3.

NOTE: In the reagent module of TALs for the 1st and 2nd source standard the concentration of each range of GRO that is potentially analyzed for are calculated based on the area count of C4-C12 in a mid point concentration being set to 100% and then each fraction is set as a percentage of C4-C12 based on its area counts. These area counts and percentages must be recalculated and reset when a new lot of 1st or 2nd source GRO standard is purchased and used.

As an example the concentration of an analyte for a given lot of standard, based off initial analysis, would be set as follows:

C4-C12 area count set to 100%

C5-C12 area count set to 99.9%

C6-C12 area count set to 87.87%

C6-C10 area count set to 77.79%

Gasoline area count set to 87.87%

7.2.4 Calibration Intermediate Standard, 200 ∝g/mL

Dilute the GRO stock standard (Section 7.2.1.1) to achieve a final GRO concentration of 200 ∝g/mL. The OKGRO stock standard is also diluted to achieve a OKGRO concentration of 200ug/mL. See Table 1, 2, and 3. (Other stock standard concentrations will require different aliquots to prepare the intermediate standard.

7.2.5 Working Level Calibration Standards

7.2.5.1 Method 8015 GRO/NWTPH and AK101 Analysis

The initial calibration is performed at 8 concentration levels for the Method 8015 GRO/NWTPH and AK101 analyses as shown in the table below. Each calibration level standard is prepared at the instrument just prior to analysis and is analyzed in the same manner as an aqueous sample (see Section 10.8). Use the following table to inject the appropriate amount of the calibration intermediate standard solution (Section 7.2.4).into 50mL of reagent water using Class A volumetric flasks. Other calibration standards can be made up based on concentrations of the intermediate stock standards and components required by the program or client. All surrogates are added by the Centurion or Archon auto sampler at a concentration of 60ug/L per calibration level.

NOTE: In the reagent module of TALs for the 1st and 2nd source standard, the concentration of each range of GRO that is potentially analyzed for is calculated based on the area count of C4-C12 in a mid point concentration standard being set to 100% and then each fraction is set as a percentage of C4-C12 based on its area counts. These area counts and percentages must be recalculated and reset when a new lot of 1st or 2nd source GRO standard is purchased and used.

As an example the concentration of an analyte for a given lot of standard, based off initial analysis, would be set as follows:

C4-C12 area count set to 100%

C5-C12 area count set to 99.9%

C6-C12 area count set to 87.87%

C6-C10 area count set to 77.79%

Gasoline area count set to 87.87%

Preparation of Working Level Calibration Standards for GRO and OK GRO Analyses

Level	Vol. of GRO or OKGRO Intermediate Working Standard (Section 7.2.4) (∝L)	Final Vol (mL)	Final GRO or OKGRO Concentration (∝g/L)	Final Surrogate Concentration (∝g/L)
1	2.5	50	10	60
2	6.2	50	24.8	60
3	12.5	50	50	60
4	25	50	100	60
5	50	50	200	60
6	125	50	500	60
7	250	50	1000	60
8	500	50	2000	60
CCV	50	50	200	60

7.2.6 Second-Source Initial Calibration Verification (ICV) and Spike Standard

NOTE: When analyzing aqueous samples, the second-source ICV working standard solution is used to prepare the laboratory control sample (LCS), which then serves the dual purpose of LCS and ICV. When analyzing soil samples, the LCS is prepared by spiking reagent sand with the ICV stock solution and extracting into methanol, then taking 1000uL of this extract and diluting in 50mL of reagent water using a 50mL Class A volumetric flask. This solution is then poured into a clean VOA vial with zero headspace and placed on the Centurion auto sampler for analysis.

Obtain a neat gasoline standard or commercially available gasoline mix standard from a source different than the source that supplied the primary standard (Section 7.2.5) and prepare according to section 7.2.2.1 and 7.2.2.2.

7.2.6.1 ICV/Spike Stock Standard Solution, 5500 ∝g/mL

If using a neat gasoline, prepare an ICV/spike stock standard solution at a concentration of 5500 ∝g/mL. Determine the density of the neat standard as described in Section 7.2.2.1 Calculate the volume of the neat standard needed to prepare 5 mL of stock standard using the following equation:

$$\text{Volume of Neat Gasoline } (\propto L) = \frac{5500(\propto g / mL) \times 5(mL)}{\rho(g / \propto L) \times 1000(\propto g / g)}$$ *Equation 3*

Dilute the calculated volume of neat standard with methanol to a final volume of 5 mL.

Alternatively, obtain a stock GRO second source standard solution in methanol from a commercial source at 5500µg/mL. The second source stock standard solution for Oklohama GRO at 10,000ug/mL is used. See Table 5 (8015 GRO, AK101 and NWTPH ID: **8015GRODCSStk**) and Table 6 (Oklahoma ID **OKGRO SS Stk**).

7.2.6.2 ICV/Spike Working Standard Solution for GRO and OKGRO

Prepare a GRO ICV/spike working standard solution by diluting the GRO ICV/spike stock standard (Section 7.2.6.1) with methanol to achieve a concentration of 99.1ug/mL. For Oklahoma GRO, dilute the OKGRO SS Stk (Section 7.2.6.1) to achieve a concentration of 100ug/mL) See Table 5 (AK101 ID **8015GRODCS**) and Table 6. (Oklahoma ID **OKGRO SS/DCS**)

7.2.7 Continuing Calibration Verification (CCV) Standard, 200 ∝g/L

7.2.7.1 The CCV for the NWTPH and 8015 GRO methods is prepared the same way the level 5 calibration standard is made. Other stock standard concentrations will require different aliquots to prepare the intermediate standard. Preparing the CCV at other concentration will require different volumes of GRO working calibration standard diluted in 50mL of DI. The OK GRO CCV can be prepared the same way a level 5 calibration standard is prepared.

7.3 Surrogate Standards

7.3.1 Surrogate Stock Standard

7.3.1.1 The surrogates for method 8015 are α,α,α-trifluorotoluene, 1-Chloro-4-flurobenzene, and Bromofluorobenzene. The stock standard for 1-Chloro-4-fluorobenzene is at 2500µg/mL. The stock standard for α,α,α-trifluorotoluene is at 2000µg/mL. The stock standard for 4-Bromofluorobenzene is at 2000µg/mL.

7.3.1.2 For 8015 and NWTPH aqueous samples, one, two or all three surrogates may be used depending on client requests. Surrogate α,α,α-trifluorotoluene is affected more by target and non-target analytes in samples.

7.3.1.3 When soil samples are preserved in the field with surrogated methanol (as recommended in Alaska Method AK101), a sample of the surrogated methanol (a field blank) must be submitted to the lab with the samples so that the surrogate concentration may be verified. In these cases, the lab may opt to add a second, different surrogate at the time of analysis to assess the analytical recovery. Depending on what surrogate is used during sampling the lab with either add α,α,α-trifluorotoluene or bromofluorobenzene.

7.3.1.4 For AK101 aqueous samples, both α,α,α-trifluorotoluene and bromofluorobenzene are used as surrogates (bromofluorobenzene Ultra Scientific STS110N). (**AK101BFBStk**)

7.3.2 Surrogate Working Standard, 60 ∝g/mL

A working surrogate solution is prepared for each surrogate by diluting stock standard solutions (Section 7.3.1) of each of the surrogates to achieve a final concentration of 60 ∝g/mL. The surrogates are combined in a single solution.. Alaska Method AK101 requires the addition of a methanol solution containing the surrogate at a concentration 2.4 ∝g/mL to the soil samples in the field. This is referred to as "surrogated methanol." A client submitting soil samples preserved in the field in this way must also provide a field blank containing the surrogated methanol so that the surrogate concentration may be verified at the time of analysis. All surrogates are added to aqueous and soil samples at the time of analysis by the Centurion auto sampler. The auto sampler adds 5µL of surrogate working standard to 5mL of sample to achieve a final on column concentration of 60µg/L.

7.3.3 Retention Time (RT) Reference Standards

Retention time reference standards are used to establish retention time windows for the analytes of interest for 8015 GRO, NWTPH, and AK101. Two sets of markers are included in the retention time marker: Alkane markers and non-Alkane hydrocarbon markers.

> **NOTE:** The Oklahoma Method 8020/8015 (modified) does not use a separate RT reference standard. The retention time windows are updated using the first CCV of the run since the retention time compounds are present in the CCV.

The following compounds are included in the Retention Time Marker stock at approximately 2000µg/mL:

Methyl-tert-butyl ether, 2-Methylpentane, 1,2,4-Timethylbenzene, Naphthalene, Pentane (C5), n-Hexane (C6), n-Octane (C8), n-Decane (C10), Dodecane (C12) and Tridecane (C13)

7.3.3 Working RT Standard (Method 8015) and OKGRO

Dilute 650∝L of the alkane and non-alkane RT stock standards (Section 7.3.3) with methanol to a final volume of 25mL to achieve a concentrations of 52µg/mL for a Working RT standard solution. A RT standard or CCVRT is made by taking 50uL of the Working RT Standard solution in 50mL of DI water and purging 5mL. All surrogates are also analyzed in this RT standard. For OKGRO, the RT standard is the initial CCV as the markers for OKGRO are included in the calibration solution.

7.4 QC Samples

NOTE: In the reagent module of TALs for the 1st and 2nd source standard the concentration of each range of GRO that is potentially analyzed for are calculated based on the area count of C4-C12 in a mid point concentration being set to 100% and then each fraction is set as a percentage of C4-C12 based on its area counts. These area counts and percentages must be recalculated and reset when a new lot of 1st or 2nd source GRO standard is purchased and used.

As an example the concentration of an analyte for a given lot of standard, based off initial analysis, would be set as follows:

C4-C12 area count set to 100%

C5-C12 area count set to 99.9%

C6-C12 area count set to 87.87%

C6-C10 area count set to 77.79%

Gasoline area count set to 87.87%

Preparation of the individual instrument and batch QC samples by method are listed in Tables 16-19.

7.4.1 Spiking LCS/LCSD and MS/MSD for Aqueous Samples

For aqueous sample batches, the LCS and LCSD are prepared by injecting 50∝L of the 100 ∝g/mL ICV/spike working standard GRO or OKGRO solution (Section 7.2.6.22) into reagent water in a 50mL Class A volumetric flask to a final volume of 50 mL. The MS and MSD are prepared by injecting 100 ∝L of the 100 ∝g/mL ICV/spike working standard solution into an aliquot of the selected aqueous sample in a 100mL Class A volumetric flask for a final volume of 100 mL. This results in a sample GRO concentration of 100 ∝g/L in the LCS, and a "spike added" GRO concentration of 100 ∝g/L in the MS and MSD.

7.4.2 Spiking LCS/LCSD and MS/MSD for Soil Samples

For soil sample batches the LCS and LCSD are prepared by spiking 5∝L of the ICV/spike stock standard solution (Section 7.2.6.1) to 5g of reagent sand extracted into 5mL of methanol. The MS and MSD are prepared similarly by adding 5∝L of the ICV spike stock standard to two separate 5g aliquots of the selected sample and extracting with 5mL of Methanol. This results in a sample GRO concentration of 4.5mg//Kg in the LCS, and a "spike added" GRO concentration of 4.5mg//Kg in the MS and MSD. For AK101 samples a spike volume of 5∝L is used for a 5mL aliquot of methanol extract

.

7.5 The stock and intermediate level standards are stored in Teflon-sealed screw-cap/crimp cap bottles with no headspace, and kept at –10 ℃ t o –20 ℃ protected from light.

7.5.1 The shelf life is 6 months after opening for the stock standards, 6 months for intermediate level standards and for the working level standards, which are prepared fresh weekly or more frequently as results indicate.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water Samples

8.1.1 Aqueous samples should be collected in triplicate without agitation and without headspace in contaminant-free glass 40 mL vials with Teflon-lined septa in the caps. The Teflon layer must contact the sample (zero headspace).

8.1.1 Analysts will choose a VOA vial without a red sticker, or without headspace > 6mm, for analysis (see DV-QA-0003 for further information). If all vials have red stickers then the VOA vials with the smallest headspace/bubbles will be chosen for analysis. Any VOA vials analyzed with a red sticker (headspace > 6 mm) will be documented in an NCM.

8.1.2 Preservation

8.1.2.1 Samples are preserved with 200 ∝L of 50% HCl. Specific method requirements are listed below:

Method	Preservation requirement
Alaska Method AK101	200 ∝L of 50% HCl
Oklahoma	Preservation with HCl to a pH<2
8015	Preservation with HCl to a pH<2
NWTPH	Preservation with HCl to a pH<2

8.1.2.2 Samples must be stored at ≤ 6℃ (without freezing) and analyzed within 14 days of collection. Unpreserved samples require analysis within 7 days.

8.1.3 Trip Blanks

8.1.3.1 Alaska Method AK101 specifies a trip blank consisting of a contaminant-free amber glass 40 mL vial with Teflon-lined septum filled to zero headspace with purged, organic-free water preserved with the same acid as the samples.

8.1.3.2 Oklahoma GRO method requires that a trip blank must be included with each sample set. If one is not provided by the client the laboratory must make note of this in the final report.

8.2 Soil Samples

8.2.1 This procedure is designed to work with soil samples preserved and extracted in methanol. This minimizes the loss of volatiles during handling in the field and in transit to the laboratory.

8.2.1.1 The Oklahoma method requires that high level samples are preserved in the field by adding 5.0 ± 0.5 g of soil to a pre-weighed 40 mL vial containing 5 mL of methanol.

8.2.1.2 Alaska Method AK101 requires preservation of soil samples in the field by submerging the sample in methanol to which the surrogate has been added. See Appendix A for detailed instructions for Method AK101.

8.2.2 If the methanol preservation cannot be done in the field, the lab must be supplied with an intact portion of sample in an EnCore™ sampler or in a volatile organic vial. In which case, the laboratory will perform the preservation step. The maximum holding time between collection and preservation at the laboratory is 48 hours for EnCore™ samples. Per Method AK101, data from lab preserved samples must be reported as "greater than or equal to" the calculated concentration of GRO as gasoline.

8.2.2.1 The lab must also be provided with a 2-ounce glass jar containing a portion of the sample to use for screening and for moisture determination.

8.2.2.2 Samples must be stored at ≤ 6 ℃ and, once preserve d in methanol, analyzed within 14 days of collection.

8.2.3 Alternatively, soil samples may be collected in 4-ounce jars packed with minimum head space and extracted with methanol at the lab and analyzed within 14 days per Method 5035 high concentration soil method. NWTPH_Gx requires that the sample be collected in VOA vials.

8.2.3.1 Method NWTPH-Gx requires that a soil extract is only good for 1 week after extraction.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation Batch

9.2.1 A preparation batch is a group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a laboratory control sample (LCS), a matrix spike (MS), and a matrix spike duplicate (MSD). Water batches should be kept together on the same instrument through the whole analytical process. Soil batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. A method blank must be run on each instrument. See DV-QA-003P for further details. For DoD QSM 4.2 or QSM 5.0, the MS/MSD must be from the project site (typically the sample to be spiked is indicated by the client) and if insufficient sample is available to analyze the MS/MSD, this is documented in an NCM and a LCSD is performed in its place. Likewise the analyst should inform the project manager, through

an NCM, if the MS/MSD is not specified by the DoD client but is prepared using another sample from another client's project.

9.2.1.1 Method NWTPH_Gx batches can only have a batch size of 10 client samples and requires that one sample duplicate be prepared with every batch. Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents.

9.2.1.2 Oklahoma requires that duplicate spikes must be prepared with every batch of ten samples or fewer.

9.2.2 As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for water batches consists of 50mL of reagent water, and for solids batches, 5g of reagent sand.

Acceptance Criteria: The result for the method blank must be less than one-half the reporting limit or less than 10% of the GRO concentration found in the associated samples, whichever is higher. For DoD QSM 4.2 or QSM 5.0 the acceptance criteria is no analytes detected >1/2 the RL (i.e. LOQ) or >1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If GRO was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. See Figure 1 in SOP DV-QA-003P for further evaluation.

9.4 Laboratory Control Sample

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of 50mL of reagent water to which 50∝L of the ICV/spike working standard is added. AK101 requires a LCSD. For soil sample batches, the LCS and LCSD consist of 5g of reagent sand to which 5∝L of the ICV/spike stock standard is added. The LCS and LCSD are carried through the entire analytical procedure.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits.

Method	LCS Acceptance Limits

Alaska Method AK101	See Table 12
Oklahoma	Water: 80-120% Soil: 60-140%
8015	Historical Limits
NWTPH	Historical Limits
DOD QSM	DOD Limits

Corrective Action: If LCS recoveries are outside of the established control limits, the system is out of control and corrective action must occur. If recoveries are above the upper control limit and GRO is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. See Figure 2 in SOP DV-QA-003P for further evaluation.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD

9.5.1 One MS/MSD pair must be processed with each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. (See Section 9.5 for spike amounts.) A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Some programs will allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

9.5.2 For DoD QSM 4.2, 5.0 or 5.1, the MS/MSD must be from the project site (typically the sample to be spiked is indicated by the client) and if insufficient sample is available to analyze the MS/MSD, this is documented in an NCM and an LCSD is performed in its place to monitor batch precision. Likewise the analyst should inform the project manager, through an NCM, if the MS/MSD is not specified by the DoD client.

Acceptance Criteria: The laboratory uses the LCS limits to evaluate recovery results for the MS and MSD. The RPD between the MS and MSD must be less than the established RPD limit, which is set at 3 standard deviations above the historical mean.

Method	**MS/MSD Acceptance Limits**
Alaska Method AK101	See Table 12
Oklahoma	Water: 80-120%

	Soil: 60-140%
8015	Historical Limits
NWTPH	Historical Limits
DOD QSM	DOD Limits

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE 1: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE 2: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.6 Surrogate Spikes

9.6.1 The surrogate standards are added to all field and QC samples by the Centurion and Archon autosamplers prior to analysis. The Centurion adds 5µL of the surrogate working solution to a 5mL aliquot of sample purged. The Archon will add 1µL of a surrogate working solution to achieve a 50µg/L final concentration of surrogates. Depending on the client surrogate requested, one, two or all three surrogates can be reported..

9.6.1.1 For AK101 aqueous samples, 5∞L of the surrogate working spike solution with BFB are added to 5mL of the aqueous sample by the autosampler just prior to purging.

9.6.1.2 The AK101 soil samples arrive at the laboratory preserved in surrogated methanol. The surrogate (either TFT or BFB) that is not in the surrogated methanol will be added by the laboratory. See WI-DV-0040 for specific spiking volumes.

9.6.1.3 For soil samples, 5∝L of the surrogate stock standard is added by the autosampler to 5mL of the diluted sample that has been extracted with methanol.

9.6.2 No surrogate is specified for the Oklahoma method so the procedure for method 8015 is followed.

NOTE: When soil samples are preserved in the field with surrogated methanol (as recommended in Alaska Method AK101), a sample of the surrogated methanol (a field blank) must be submitted to the lab with the samples so that the surrogate concentration may be verified. In these cases, the lab may opt to add a second, different surrogate at the time of analysis to assess the analytical recovery. Typically the lab uses the surrogate 1-chloro-4-fluorobenzene to monitor recovery.

Acceptance Criteria: The recovery of the surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Method	**Surrogate Acceptance Limits**
Alaska Method AK101	See Table 12
Oklahoma	Water: 75-125% Soil: 75-125%
8015	Historical Limits
NWTPH	Historical LImits
DOD QSM	DOD Limits

Corrective Action: If surrogate recoveries are outside the established limits, verify calculations, dilutions, and standard solutions. Also verify that instrument performance is acceptable. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem. A low surrogate recovery in the method blank will invalidate the method blank and all samples will require re-extraction to prove that there is not a low bias in the analytical QC batch. A high surrogate in the method blank may be acceptable if the MB has no detections. In either case check the project requirements for the appropriate corrective action.

If the surrogate recovery is outside of the established limits due to well documented matrix effects, the results must be flagged and an explanation included in the

report narrative. As with matrix spike failures, some programs may require additional analyses to confirm suspected matrix interferences. High surrogate failures and samples with no detections may be narrated. The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to re-prepare / reanalyze a sample once to demonstrate a matrix effect is reproducible.

For Alaska Method AK101 soils that are preserved in the field with surrogated methanol, if the field-added surrogate fails acceptance criteria, it may be possible to report results with appropriate narration if the laboratory-added surrogate meets acceptance criteria.

If the surrogate peak falls outside of its established RT window, it may be necessary to re-establish the center of the RT window and reanalyze affected samples. This requires re-starting the analytical sequence. (See Section 10.6.)

See Figure 3 in SOP DV-QA-003P for further evaluation.

9.7 RT Reference Standard (Method 8015):

A RT reference standard containing the predominant gasoline components (see Section 7.3.3) is analyzed with each initial calibration to establish the retention time window. The RT reference standard is prepared by adding 50∝L of the working RT standard (Section 7.3.3) to 50mL of reagent water. The Centurion autos sampler will purge 5mL along with the surrogates into the purge and trap apparatus. For the Archon auto sampler, 5uL of the working RT standard is added to 5mL of reageant water and purged.

9.8 RT Marker Standard (AK101):

9.8.1 At the beginning of each day's analytical sequence, a RT reference standard is analyzed. The surrogates are added to the RT reference standard and purged. The midpoint of the RT window for the 1-Chloro-4-Fluorobenzene is set for the day based on these results. If the midpoint of the RT window for the 1-Chloro-4-Fluorobenzene in any subsequent field sample, QC sample or CCV slips by more than the established retention time window, then the data for that sample or standard is invalid and samples are reanalyzed as necessary.

9.8.2 The RT standards are analyzed prior to any samples. The surrogate retention times are set in the CCVRT.

NOTE: Oklahoma GRO method does not use a separate RT marker standard as all the pertinent markers are present in the CCV. .

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 It is recommended that samples be screened prior to analysis by purge-and-trap GC/FID. Sample aliquots used for the definitive analysis are based on the screening data.

10.4 Gas Chromatographic Conditions

10.4.1 Recommended instrument conditions are shown in Table 9. These can be adjusted prior to calibration in order to improve performance.

10.4.2 The routine purge volume is 5 mL.

10.5 Retention Time (RT) Window

10.5.1 Before establishing retention time (RT) windows, ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

10.5.2 Analyze an RT Reference Standard (Section 7.5) three times throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

10.5.3 Calculate the mean and standard deviation of the three absolute retention times for each surrogate, the internal standard, and any single component markers.

10.5.3.1 The width of the retention time window for each analyte and surrogate peak is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period.

10.5.3.2 In those cases where the standard deviation for a particular analyte is zero, the minimum window to use is method specific:

10.5.3.2.1 8000B or 8000C: ± 0.03 minute

10.5.3.2.2 AK101: ± 0.05 minute

10.5.3.2.3 Oklahoma: ± 0.1 minute

10.5.4 For the GRO identification and quantization, the retention time window is specified by the method based on the range of hydrocarbons to be included as described below:

10.5.4.1 SW-846 Methods (8015B, 8015C and 8015D):

C_6-C_{10}: use the peak start time for 2-methylpentane through the peak end of 1,2,4-trimethylbenzene.

C_6-C_{12}: use the peak start time for 2-methylpentane through the peak end of C_{12} (n-dodecane).

C_4-C_{12}: the range begins immediately following the elution of the methanol solvent peak and continues until the beginning of C_{13} (tridecane) peak.

Gasoline: use the peak start time for 2-methylpentane through the peak end of 1,2,4-trimethylbenzene. .

10.5.4.2 AK101:

C_6-C_{10}: start of C_6 through start of C_{10}.

10.5.4.3 NWTPH-Gx:

Peak start time for 2-methylpentane through the peak end time of C_{12} (n-dodecane).

10.5.4.4 Oklahoma GRO:

Start approximately 0.1 min before the retention time of MTBE and ending 0.1 min after the RT of naphthalene.

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10.5.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or after any major adjustment to the instrument conditions. The data must be retained by the laboratory.

10.6 Daily Retention Time Windows

10.6.1 At the beginning of each daily analytical sequence, analyze the RT marker standard (Section 7.1). The center of the retention time windows are adjusted based on the retention time observed for the RT marker standard and the start and endpoints for the hydrocarbon ranges.

10.6.2 The standards used for retention time verifications are documented in chrome.

10.6.3 The Oklahoma Methods 8020/8015 (modified) does not use a separate RT reference standard. The retention times are updated using the first CCV of the run.

10.7 Analytical Sequence

10.7.1 The analytical sequence starts with an RT reference standard, initial calibration (ICAL) and initial calibration verification (ICV or LCS), or with a daily continuing calibration verification (CCV). .

10.7.2 After the opening CCV an instrument blank must be analyzed to demonstrate that the instrument and reagents are free of any interferences. For waters and soils the instrument blank is reagent water. The batch method blank can also be used to demonstrate the system is free of interferences.

10.7.2.1 For AK101 an instrument blank must be run with each analytical batch (or daily, whichever is more frequent).

10.7.3 The LCS and the method blank are analyzed following the initial calibration verification or the daily CCV.

10.7.4 Samples are analyzed and are interspersed with a CCV check performed after every 12 hours or every 10 samples, whichever is more frequent.

10.7.4.1 For Oklahoma GRO, the appropriate instrument blank must be analyzed before the analysis of every CCV check to demonstrate that the system is free of interferences and carryover. If an instrument blank exceeds ½ the RL then the entire bracket must be reanalyzed.

10.7.5 If the measured concentration of any sample exceeds the highest calibration standard concentration, the sample extract must be diluted and reanalyzed. All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

10.7.6 Blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination beyond established "column bleed", the column must be baked out and subsequent blanks analyzed until the system is shown to be free from contaminants.

10.7.7 If a sample is discovered to have a high concentration, then the sample immediately following the high concentration sample must be evaluated for

detection of target analytes. If target analytes are detected at or above the RL, then reanalyze the sample to rule out carryover from the high concentration sample unless some other objective evidence indicates that reanalysis is not necessary.

10.7.8 The analytical sequence is bracketed with a closing CCV.

10.8 Aqueous Sample Analysis

10.8.1 Unless a manual or serial dilution needs to be performed, all samples are analyzed without opening the 40mL VOA vial and placing on the Centurion analyzer after allowing sample to come to room temperature. Once purged and analyzed by the Centurion, the sample vial can not be re-sampled as all GRO is effectively purged from the sample..

10.8.2 Surrogates are added by the Centurion immediately prior to the start of purging. Serial dilutions in the following increments can be automatically performed by the Centurion auto sampler using reagent water and the auto sampler syringe: 2x, 4x, 5x, 10x, 20x, 40x, 50x, 100x, 200x, 400x

10.8.3 Add 50∝L of the ICV/spike working standard (Section 7.2.6.2) to the LCS, MS, and MSD.

10.8.4 This process destroys the validity of the liquid sample for future analysis. Therefore, if there is only one 40 mL vial, then transfer the remaining sample from the 40 mL vial to a 20 mL vial so that it is completely full (no headspace). This second sample is maintained only until it has determined that the first sample has been analyzed properly. If a second analysis is needed, it must be analyzed within 24 hours. Care must be taken to prevent air from getting into the 20 mL vial.

10.9 Soil Sample Preparation

NOTE: Alaska Method AK101 and NWTPH_Gx require results for soil samples to be reported on a dry-weight basis. Typically 8015 is reported on a dry weight basis, at the discretion of the client. Moisture determination must accompany all soils data. The client must provide an unpreserved portion of sample for the moisture determination. The percent moisture is determined by weighing an aliquot of the sample as received and weighing it again after it has dried overnight at ~105ϒC. Oklahoma is reported on a wet-weight basis.

10.9.1 Preserved Soil Samples Received in Pre-Weighed Containers

NOTE: In the field, the soil sample is placed in a vial that contains surrogated methanol. The weight of the vial plus methanol is determined prior to adding the sample and is marked on the vial.

10.9.1.1 Weigh the sample container and subtract the number written on the vial from this weight to obtain the weight of the sample. Do not add any labels to the vial until this weight has been determined.

NOTE: For AK101 the number marked on the vial is the typically the weight of the vial and does not include the weight of the methanol. It is recommended to mark and date the meniscus for the methanol on the outside of the jar. It is also recommended that the samples equilibrate for at least 48 hours after sampling.

10.9.1.2 Surrogates for soils will be added by the auto sampler to all the samples at the time of purging.

NOTE: For Alaska Method AK101 samples, the methanol used to preserve the sample in the field should already contain the appropriate surrogate, either bromofluorobenzene or α,α,α-trifluorotoluene. A second, alternate surrogate should be added in the laboratory to verify analytical accuracy and serve as check for the field-added surrogate.

10.9.1.3 Add the designated amount of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.9.1.4 Proceed to Section 10.10.1

10.9.2 Unpreserved Soil Samples Received in Pre-Weighed Containers

10.9.2.1 Weigh the sample container as received to confirm the field weights, and to determine the weight of the sample, and record the weight.

10.9.2.2 If the field weight differs by more than 0.2 gram from the laboratory-determined weight, the sample collection is considered invalid. The most common reason for disagreement in weights is extra labels added to the container after weighing in the field. However, it is possible that the weights disagree because volatile components have been lost since the time of sample collection. Contact the client before proceeding with analysis if the weights do not agree.

10.9.2.3 If the weight of the sample is ≥ 10 g, then treat the sample as "not pre-weighed" and proceed to Section 10.9.3

10.9.2.4 If the weight of the sample is less than 10 g, then add the designated amount of methanol and specific surrogate stock standard that has been specified in method or project requirements (Section 7.3.1) to each field sample, blank, LCS, MS, and MSD.

10.9.2.5 Add the designated amount of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.9.2.6 Proceed to Section 10.10.

10.9.3 Unpreserved Soils Received in Containers <u>Not</u> Pre-Weighed

10.9.3.1 EnCore™ or Core-N-One™ Soil Samples: Obtain the tare weight of a 20 mL vial including the cap and that contains 5 mL of methanol. Extrude the sample from the EnCore™ into the vial, cap, and weigh.

10.9.3.2 Add the designated amount of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.9.3.3 If there is sufficient sample, split the sample to create a duplicate portion and extract both portions of the sample.

NOTE: To comply with Method 5035, this extraction must be done within 48 hours of collection. Some work plans may have special provisions for longer holding times for samples frozen in EnCore™ or Core-N-One0™ samplers, but that is not routine.

10.9.3.4 **Bulk Soils**: Bulk soil samples are submitted in packed, unweighed jars, vials, or core tubes. Transfer 5 ± 0.5 grams of soil into a prepared 20 mL vial. Immediately add 5 mL of methanol and cap the vial. This transfer must be performed very quickly to minimize loss of target analytes.

10.9.3.4.1 For NWTPH_Gx samples transfer approximately 5 grams of soil into a prepared 20 mL vial and add 5 mL of methanol.

10.9.3.5 Add the designated amount of the ICV/spike stock standard solution (Section 7.2.6.1) to the LCS, MS, and MSD.

10.10 Soil Sample Analysis

10.10.1 After combining the sample and methanol, add the spikes (if appropriate) through the septum of the cap for the LCS, LCSD, MS, and MSD. For specific spiking instructions see WI-DV-0040 GC VOA Spike Guide.

10.10.1.1 For method 8015 GRO, NWTPH_Gx and Oklahoma samples, shake samples for two minutes or put them on a shaker table for a comparable amount of time.

10.10.1.2 For AK101 samples swirl the jars gently for two minutes.

10.10.2 Allow the sediment to settle.

For AK101 samples do not centrifuge, filter or decant any of the samples. The entire contents of the sample container must be represented in the analysis. If the sample aliquot has a large amount of solids make a note of it.

10.10.3 If not analyzed immediately, these extracts must be stored at ≤ 6℃ in the dark.

10.10.4 Use Table 11 to determine the volume of methanol extract to add to 50 mL of reagent water for analysis. If a screening procedure was used, use the estimated concentration to determine the appropriate methanol volume. For

analysis on the Centurion auto sampler, the maximum and default volume of methanol that can be added to the reagent water for analysis is 1000∝L for a 50x dilution of the soil extract. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. After adding the methanol extract to 50mL of reagent water, transfer the solution to a clean 40mL VOA vial so that there is zero headspace to be placed on the Centurion auto sampler. Dilutions can be made either externally by adding a volume less than 1000µL to 50mL of reagent water, or by taking the default 50x dilution and having the auto sampler perform a diluition. The MB, LCS, and LCSD will be prepared by adding 1000µL of the extract to 50mL of reagent water and transferring to a 40mL vial without headspace.

10.10.5 5∝L of the Surrogate working solution (Section 7.3.2) is added to each 5mL of sample purged by the Centurion auto sampler.

10.10.6 For instruments using an Archon 4552 autosampler, the samples are prepared in 40 mL VOA vials using 5mL of reagent water. The sealed VOA vials are placed in the autosampler. The method blank, LCS and LCSD should contain 5g of laboratory sand. The auto sampler will add 1µL of surrogate working standard to achieve a 50µg/L final concentration of surrogates.

10.10.7 Analyze all method blanks on the same instruments that are used for the samples. For the Centurion, the method blank should contain 1000∝L in 50mL of reagent water of the methanol used to extract the organic-free reagent sand.

10.11 Instrument Troubleshooting and Maintenance

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance.

10.11.1 If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control.

10.11.2 Any auto sampler ports that had high level samples analyzed in the previous run should be cleaned and baked out as needed. A blank should be run on the port to ensure that any carryover has been removed.

10.11.3 A reduced response may indicate that the system needs to be evaluated for leaks.

10.11.4 Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed.

10.11.5 Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

10.12 Calibration

Detailed information regarding calibration models and calculations can be found in Corporate SOP **CA-Q-P-003** *Calibration Curves and the Selection of Calibration Points* and in the public folder, Arizona Calibration Training.

10.12.1 Initial Calibration (ICAL)

10.12.1.1 A new calibration curve must be generated after major changes to the system or when continuing calibration criteria cannot be met. Major changes include installation of new columns and changing FID jets.

10.12.1.2 The ICAL is performed using six to eight concentration levels (see Section 7.2.5 and Tables 5-7). The lowest calibration concentration is at or below the laboratory reporting limit (RL) concentration. The highest standard defines the highest sample extract concentration that may be reported without dilution.

10.12.1.3 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.12.1.4 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if <u>all</u> of the following conditions are met:

10.12.1.4.1 The rejected point is the highest or lowest on the curve, i.e., <u>the remaining points used for calibration must be contiguous</u>;

10.12.1.4.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.12.1.4.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.12.1.4.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.12.2 The external standardization method is used for all methods except Oklahoma which uses the internal standardization method for the BTEX compounds. Tabulate the area response for each calibration level against the concentration injected. The ratio of the response to the concentration injected, defined as the calibration factor (CF), is calculated for the standard at each concentration as follows:

$$CF_i = \frac{A_{fuel}}{C_{fuel}}$$ *Equation 4*

Where:

CF_i = Calibration factor for the ith calibration level.

A_{fuel} = Total area of the fuel calibration standard peak.

C_{fuel} = Concentration of fuel calibration standard, mg/mL

If the percent relative standard deviation (%RSD) for the average (mean) of the calculated calibration factors is less than 20%, the average calibration factor can be used for sample quantitation.

$$\text{AverageResponseFactor} = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ *Equation 5*

Where:

CF_i = Calibration factor for the ith calibration level.

n = The number of calibration levels.

10.12.3 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through other options until the calibration criteria are met. Pay particular attention to the residuals noticed at the upper and lower end of the curves. This may be cause for rejection of a curve fit even if the calibration acceptance criteria are met.

10.12.3.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte or hydrocarbon range in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in Equation 4 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$ *Equation 6*

Where:

CF_i = Calibration factor for the ith calibration level.

n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\%$$ *Equation 7*

Where SD is the standard deviation of the average CF, which is

calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(CF_i - \overline{CF}\right)^2}{n-1}}$$ *Equation 8*

10.12.3.2 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The % RSD must be ≤ 20% (AK101 upper limit is 25%).

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH_Gx) of the expected concentration for that point.

Corrective Action: If the % RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.12.3.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The weighting used is the reciprocal of the square of the standard deviation. The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b$$ *Equation 9*

Where:

y = Instrument response (peak area or height).

x = Concentration of the target analyte in the calibration standard.

a = Slope of the line.

b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 10}$$

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 11}$$

Where:

A_s = Area of the chromatographic peak for the target analyte in the calibration standard.

A_e = Area of the chromatographic peak for the target analyte in the sample extract.

a = Slope of the line as determined by the least-squares regression.

C_s = Concentration of the target analyte in the calibration standard.

C_e = Concentration of the target analyte in the sample extract.

b = Intercept of the line as determined by the least-squares regression.

10.12.3.4 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of a weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Also examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the

residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., DoD) require a correlation coefficient ≥ 0.995. DoD QSM 5.0 requires the coefficient of determination (r^2) to be >0.99. Note that the AK102/AK103 method requires that r^2 be greater than or equal to 0.995 but method AK101 does not list a specification.

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH) of the expected concentration for that point.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.12.3.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 12}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.12.3.6 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination must be ≥ 0.990.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica-Denver.

10.12.3.6.1 Second-order regressions should be the last option. Note that some programs (e.g., South Carolina and

method NWTPH_Gx) do not allow the use of second-order regressions.

10.12.3.6.2 Before selecting a second-order regression calibration model, it is important to ensure the following:

- ∞ The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- ∞ The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- ∞ The distribution of concentrations is adequate to characterize the curvature.

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH) of the expected concentration for that point.

10.12.4 Internal Standard Calibration

10.12.4.1 The ratio of the peak area of the target compound in the sample extract to the peak area of the internal standard in the sample extract is compared to a similar ratio derived for each calibration standard. This ratio is the response factor (RF).

For each of the initial calibration standards, calculate the RF value for the GRO relative to the internal standard as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$ *Equation 13*

Where:

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Absolute weight of analyte purged in ∝g.

C_{is} = Absolute weight of the internal standard purged in ∝g.

10.12.4.2 The response factor is a measure of the slope of the calibration relationship and the assumption is made that the curve passes through the origin. To evaluate the linearity of the calibration, calculate the mean response factor, the standard deviation (SD), and the relative standard deviation (RSD) as follows:

$$\text{mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$ *Equation 14*

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}}$$ *Equation 15*

$$RSD = \frac{SD}{\overline{RF}} \times 100\%$$ *Equation 16*

Where:

RF_i	=	Response factor for the ith calibration level.
n	=	Number of calibration levels

If the RSD of the response factors is ″ 20%, then linearity through the origin may be assumed, and the average response factor may be used to determine sample concentrations as follows. Otherwise, a linear least-squares regression fit must be used for the calibration.

$$C_e = \frac{A_s \times C_{is}}{A_{is} \times \overline{RF}}$$ *Equation 17*

Where:

C_e	=	Absolute weight of the analyte or surrogate in the sample purge in ∝g.
A_s	=	Peak area of the analyte or surrogate.
A_{is}	=	Peak area of the internal standard.
C_{is}	=	Absolute weight of the internal standard purged in ∝g.
$\overline{RF}$	=	Average response factor from calibration.

10.12.4.3 To establish the least-squares regression calibration function, the instrument response (peak area) is treated as the dependent variable (y), and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = mx + b$$ *Equation 18*

Where:

y	=	Instrument response (peak area).
m	=	Slope of the line.
x	=	Concentration or amount of the calibration standard.
b	=	Intercept of the line.

10.12.4.4 To calculate a sample concentration or amount, the equation is solved for concentration/amount, taking the following form:

$$x = \frac{y - b}{m}$$ *Equation 19*

For the internal standard method, the regression equation is rearranged as follows:

$$\frac{A_s C_{is}}{A_{is}} = mC_s + b$$ *Equation 20*

To calculate a sample concentration (or amount), this equation is solved for concentration (or amount) as follows:

$$C_e = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{m}$$ *Equation 21*

Where:

C_e = Concentration or amount of the target analyte in the sample purge.

A_s = Peak area for target analyte in sample purge.

A_{is} = Peak area for internal standard.

C_{is} = Concentration or amount of the internal standard.

m = Slope of the line.

10.12.4.5 The correlation coefficient, r, of the fitted line must be ≥ 0.990. Some programs (e.g., DoD) require r ≥ 0.995, unless approval is given in the project QAPP to use 0.990.

10.12.4.6 If the ICAL %RSD or correlation coefficient linearity criteria are not met, sample analysis cannot be performed. Check that the instrument is performing properly, and adjust as needed. Check that the standards are made correctly. After correcting any problems, prepare and reanalyze a new ICAL.

10.12.5 Second-Source Initial Calibration Verification (ICV)

Analyze a 100 ∝g/L second-source verification standard after each ICAL. For aqueous sample batches, the ICV is also the LCS (Section 7.2.6.2). For soil sample batches, the ICV is separate from the soil LCS. (For AK101 calibrations also run a instrument blank after each ICAL.)

Acceptance Criteria: The percent difference (%D) between the measured concentration for this standard and the expected concentration must be within ± 15%. When the ICV also functions as the LCS for aqueous soil batches, the percent recovery for the LCS must be within 85 to 115%. Methods 8015C and 8015D allow up to ± 20%D.

Corrective Action: If the %D or % recovery for the second-source verification fails acceptance criteria, then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result also fails acceptance criteria, then re-prepare the verification standard, and/or re-prepare and rerun the ICAL.

The %D and % recovery are calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Expected Conc}}{\text{Expected Conc}} \times 100\% \qquad \textit{Equation 22}$$

$$\%\,\text{Recovery} = \frac{\text{Conc Recovered}}{\text{Conc Added}} \times 100\% \qquad \textit{Equation 23}$$

10.12.6 Continuing Calibration Verification (CCV)

Analyze a mid-point calibration standard every 12 hours or after 20 sample analyses, whichever is more frequent, and upon completion of sample analyses to bracket the run. Method 8000C requires two levels for the CCV when a quadradic fit is used for the ICAL. The concentrations of the CCVs in this case are near the midpoint of the calibration curve and near the RL. (See Section 7.2.2) It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

Acceptance Criteria:

Method	CCV Acceptance Limits
Alaska Method AK101	± 25%
Oklahoma	± 20%
8015B	± 15%

Method	CCV Acceptance Limits
8015C, 8015D	± 20%
NWTPH	± 20%
DOD QSM	± 20%

Corrective Action: If one or more analytes fails the acceptance criteria, check the instrument operating conditions, and if necessary, restore them to original settings or perform routine maintenance. This may include clipping the column, changing the liner, or other minor instrument adjustments. Inject another aliquot of the CCV standard.

If the response for any analyte is still not within the respective acceptance limits, then a new initial calibration must be performed. Any samples injected after the last valid CCV standard must be re-injected. DoD QSM 5.0 requires recalibration and reanalysis of all affected samples since the last acceptable CCV. As an alternative, the laboratory may analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, samples may be reported without reanalysis. If either fails, take corrective actions and recalibrate the instrument: then reanalyze all affected samples since the last acceptable CCV.

If a DoD client accepts TestAmerica's Technical specifications for DoD QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags requires by the project.

11.0 Calculations / Data Reduction

The concentration of 8015 GRO in the sample is determined by calculating the absolute weight of analyte purged, from a summation of peak response for all chromatographic peaks, resolved and unresolved, eluting between the method required start and end of the envelope as listed below.

Quantitation Range by Method

Method	***All chromatographic peaks, resolved and unresolved, eluting between:***
Alaska Method AK101	the peak start time of hexane (C_6) and the peak start time of decane (C_{10}).
Oklahoma	0.1 min before MTBE and 0.1 min after naphthalene.
8015	peak start of 2-methylpentane (C_6) and the peak end of 1,2,4-

Method	***All chromatographic peaks, resolved and unresolved, eluting between:***
	trimethylbenzene (C_9)
NWTPH	the peak start time for 2-methylpentane (C_6) and the peak end time of C_{12} (dodecane).

Second column confirmation is not normally performed for this method as confirmation of chemical identity is not part of the analysis. *Calculation of Sample GRO Concentration*

11.1.1 GRO in the sample extract (C_e) is calculated using the calibration function as described in Section 10.12 the GRO concentration in the original sample is calculated as follows

11.1.2 GRO Concentration in Aqueous Samples

:

$$C_s = \frac{C_e}{V_s} \times DF \qquad \textit{Equation 24}$$

Where:

C_s = Concentration of GRO in the original sample in ∝g/L.
C_e = Absolute weight of GRO in sample purged in ∝g.
V_s = Volume of sample purged in liters.
DF = Dilution factor.

11.1.3 GRO Concentration in Soil Samples

$$C_s = C_e \times \frac{V_m \times DF}{W_s \times V_p} \qquad \textit{Equation 25}$$

Where:

C_s = Concentration of GRO in the original sample.
C_e = Absolute weight of GRO in sample extract purged in ∝g.
V_m = Volume of methanol extract in ∝L (e.g., 10 mL = 10,000 ∝L).
W_s = Weight of sample extracted in Kg. For Alaska Method AK101 and as specified by project requirements, the dry weight is used.
V_p = Volume of extract actually purged in ∝L.
DF = Dilution factor.

NOTE: For Alaska Method AK101, and as specified by project requirements, the percent moisture of a soil sample is calculated by subtracting the weight after drying from the original sample weight, dividing that by the original sample weight, and expressing the result as a percent.

NOTE: For work performed for the state of Alaska, the volume of methanol used for extraction must be adjusted for the moisture content of the sample by the following equation:

$$V_t = V_m + M * W_s/100 \qquad \textit{Equation 26}$$

Where:

Vt = final extract volume, corrected for moisture (mL)
Vm = volume methanol used for extraction (mL)
M = moisture content of the sample (%)
Ws = aliquot of sample extracted (g)

11.2 Upon completion of the analytical sequence, transfer the raw chromatography data to CHROM for further processing.

11.2.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.2.2 All manual integrations must be justified and documented. See DV-QA-011P requirements for manual integration

11.2.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. The integration for GRO for all methods must be baseline-to-baseline integration. For surrogates and single peak analytes the integration needs to be valley-to-valley.

11.2.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the before and after chromatograms to the same scale and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration.

11.3 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.3.1 Perform a Level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.3.2 Submit the data package and review checklist to the peer reviewer for the Level 2 review. All manual integrations must be evaluated by the peer reviewer and initialed on the manual integration summary report. The Level 2 review is documented on the review checklist initiated at the Level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Method Performance

12.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Demonstration of Capabilities Method Detection Limit Study (MDL)

12.3 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methanol extract vial waste – Expired Extract Vials, Waste Stream A

14.2.2 Purge-and-trap aqueous waste – Flammable Solvent Waste, Waste Stream C

14.2.3 Solvent rinse waste – Flammable Solvent Waste, Waste Stream C

14.2.4 Expired Chemicals/Reagents/Standards – Contact the Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

15.1.3 Method 8015B, Nonhalogenated Organics Using GC/FID, Revision 2, December 1996.

15.1.4 Method 8015C, Nonhalogenated organics by Gas Chromatography, Revision 3, February 2007.

15.1.5 Method 8015D, Nonhalogenated organics by Gas Chromatography, Revision 4, June 2003.

15.1.6 Method 5000, Sample Preparation for Volatile Organic Compounds, Revision 0, December 1996.

15.1.7 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December 1996

15.1.8 Method 5030C, Purge-and-Trap for Aqueous Samples, Revision 3, May 2003.

15.1.9 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December 1996.

15.1.10 Method 5035A, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 1, July 2002.

15.2 American Petroleum Institute "Method for the Determination of Gasoline Range Organics," Draft Revision 4- August 18, 1992, prepared by Rocky Mountain Analytical for the American Petroleum Institute.

15.3 Alaska Method AK101, "For the Determination of Gasoline Range Organics", Version 04/08/02.

15.4 Oklahoma Methods 8020/8015 (modified) Gasoline Range Organics (GRO), Revision 4.0, 02/24/96.

15.5 NWTPH-Gx, "Volatile Petroleum Products Method for Soil and Water", Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

16.0 Method Modifications:

Item	Method	Modification
1	NWTPH-Gx	NWTPH-Gx suggests the use of 1,4-difluorobenzene or bromofluorobenzene but also allows for the use of other surrogates as long as these compounds are non-polar, purgeable from water and do not coelute with any significant component of gasoline. α, α, α-Trifluorotoluene is used in place of the method suggested surrogates.
2	OK Methods	Standard soil RL of 0.5 mg/*Kg* for GRO and 0.050 mg/kg for individual components is higher than the method specified RL of 0.1 mg/*Kg* for GRO and 0.01 mg/Kg for individual components. The laboratory MDLs do not support the method RL. The Oklahoma method identifies a direct purge of soil samples unless dilution is required and then a methanol extraction is performed. The lab uses the methanol extraction for all samples.
3	OK Methods	The spike level used by the laboratory to fulfill Oklahoma's requirement for the analysis of 7 replicates for demonstration of ability to generate acceptable accuracy and precision for both water and soil have been increased to the method RL for each individual compound. The method requirement is to spike at the method reporting limit of 2 ug/L for all individual component for waters and at the method reporting limit of 10 ug/Kg for all individual components for soils. The laboratory MDLs do not support the method RLs for all compounds and the spike level used is the laboratory RL for the individual components.
4	OK Methods	For OK samples that are over instrument calibration range the samples will be diluted using the appropriate matrix for the sample. Water samples will be diluted in water and soil samples will be diluted in MeOH. The dilutions will not be performed as outline in section 9.4.2 in the source method.

17.0 Attachments

Table 1: Typical Commercial GRO/BTEX Calibration Standard
Table 2: Typical Commercial Alaska Method AK101 GRO Calibration Standard
Table 3: Typical Commercial GRO/BTEX Calibration Standard for Oklahoma Methods 8020/8015 (Modified)
Table 4: Oklahoma GRO Method Calibration Levels
Table 5: Typical Commercial GRO/BTEX Second-Source Standard

Table 6: Typical Commercial Second Source Standard for Oklahoma Methods 8020/8015 (Modified)
Table 7a: Typical RT Stock Standards
Table 7b: Typical AK101 RT Standards
Table 8: Recommended Instrument Conditions
Table 9: Preservation and Standard Prep for Medium Level GRO soils
Table 10: Amount of Methanol Extract Needed for Analysis of Soils
Table 11: Alaska Method AK101 Quality Control Acceptance Criteria
Table 12: Oklahoma GRO Analyte/Peak Number
Table 13: Method 8015 and NWTPH-Gx Instrument QC and Sample Spiking Amounts
Table 14: Method AK101 Instrument QC and Sample Spiking Amounts
Table 15: Okla_GRO Method Instrument QC and Sample Spiking Amounts
Appendix A: Sample Collection Instructions for Alaska Method AK101 GRO/BTEX
Figure 1: Sample Chromatogram for Alkane RT Marker Standard
Figure 2: Sample Chromatogram for GRO Retention time Standard
Figure 3: Sample Chromatogram for 8015 GRO CCV
Figure 4: Sample Chromatogram for AK101 CCV
Figure 5: Sample Chromatogram for AK101 CCV
Figure 6: Sample Chromatogram for OKGRO CCV on FID

18.0 Revision History

- Revision 15, dated 04 June 2020
  - Updated to match Best Practice procedures
  - Annual Review
  - Updated Copyright Information
- Revision 14, dated 05 December 2018
  - Added note sections to 7.2.7.3 and 7.6
  - Added section 8.1.1 about headspace
- Revision 13, dated 30 September 2018
  - Added section 8.1.1.1 regarding headspace/bubble check requirements by Sample Receiving and Analysts.
- Revision 12, dated 2 May 2018
  - Added Kansas LRH method throughout
- Revision 11, dated 31 March 2017
  - Added Kansas LRH method throughout
  - Reorganized and updated information in Section 1
  - Reorganized standards and spiking information to improve readability
  - Removed all references to AFCEE and use of chlorobenzene as a surrogate
  - Added requirement to analyze LCS/LCSD when MS/MSD not run in the batch for DoD samples to align with current requirements
  - Updated MS/MSD corrective actions consistent with current policy
  - Added Tables 16-19 to provide summary for spiking samples and instrument and batch QC
- Revision 10, dated 31 October 2015

- Added statement to Section 9.2 regarding new analysts working under supervision prior to approval of IDOC.
- Revised Section 9.6 to reflect current practice
- Added new Section 10.10.8 – sample prep for OI autosampler
- Removed statement regarding homogenization of bulk samples.
- Updated Section 10.12 to reference new corporate SOP number for calibration curves
- Revised Section 12 to reflect current practice
- Formatting and editorial changes throughout

∞ Revision 9, dated 30 September 2014

- Added statement to Section 2.3 that internal standard used for OK BTEX only.
- Added information regarding method blank to Section 4.2
- Added details regarding instruments and columns to Section 6.1.1 and 6.1.2
- Added TALS reagent IDs to Section 7
- Added new section 7.6.3
- Updated Section 9.1, 10.1 and added new 10.2 to reflect current practice
- Added requirements for DoD QSM 5.0 throughout
- Removed old section 10.3.3
- Added statement to section 10.5.5 regarding frequency of RT windows.
- Added statement to section 10.7.5 regarding dilutions.
- Added statement to section 10.8.5 regarding autosampler.
- Separated soil analysis from water analysis into its own section, 10.10
- Added Section 10.11, Instrument Troubleshooting and Maintenance
- Updated instrument conditions, by instrument in Table 9
- Updated Figure 6

∞ Revision 8, dated 19 August 2013

- Formatting and editorial corrections through out document
- Sections 7.2.7, 7.5, – removed references to 8021B GRO
- Changed surrogate compound from Bromofluorobenzene (BFB) to α,α,α-trifluorotoluene through out document.

∞ Revision 7.2, dated 13 August 2012

- Revised sections 1.0 and 3.5.
- Added marker compound information to section 2.3
- Added note to section 3.5.
- Added information concerning co-eluting compounds to section 4.1
- Added 8015 vs AK101 standard information to section 7.2.1.2
- Changed surrogate from α,α,α-trifluorotoluene to 4-bromofluorobenzene

∞ Revision 7.1, dated 18 July 2011

- Revised Oklahoma IDOC procedure (section 12.2.1) to increase spike level for Soil IDOC to 50 ug/kg, the laboratory RL for the individual components.
- Clarified method modification 4 and added method modification 5.

∞ Revision 7, dated 11 April 2011

- Combined with SOP DV-GC-0028, Gasoline Range Organics (GRO) by GC/FID SW846 Method 8015C to provide a comprehensive SOP that covers

GRO by Methods 8015B, C and D, 8021B and the state methods of Alaska, Oklahoma and Washington.

- Removed references to CA LUFT Manual and method
- Removed LCSD requirement except as needed based on TA-Denver policy and client request.
- Included both internal and external standard calibration discussions
- Updated formatting
- Removed grand mean of %RSD for continuing calibration acceptance criteria
- Included Oklahoma IDOC protocol
- Expanded references section to include all methods in SOP including prep methods.
- Updated tables to include each set initial calibration standards based on individual method requirements.
- Added example chromatograms for RT Marker Standard and GRO standard

Earlier revision histories have been archived and are available upon request.

TABLE 1.

TYPICAL COMMERCIAL GRO/BTEX CALIBRATION STANDARD

NOTE:	The following formulation is an example of a commercially available GRO/BTEX standard. In this case, the formulation is that of AccuStandard GA-001-20X-BTEX. The components are in a methanol solution.	
Component	**Stock Standard Concentration (∝g/mL)**	**Intermediate Standard Concentration (∝g/mL)**
Benzene	78	1.56
Ethylbenzene	320.5	6.41
Gasoline	10,000	200
m- & p-Xylenes	381.2	7.62
o-Xylene	137.7	2.75
Toluene	97.0	1.94

TABLE 2.

TYPICAL COMMERCIAL ALASKA METHOD AK101 GRO CALIBRATION STANDARD

NOTE:	The following formulation is an example of a commercially available GRO standard for the Alaska method AK101. In this case, the formulation is that of AccuStandard AK-101GCS-R1. The components are in a methanol solution.	
Component	**Stock Standard Concentration (∝g/mL)**	**Intermediate Standard Concentration (∝g/mL)**
Gasoline	744000	200

TABLE 3.

TYPICAL COMMERCIAL GRO/BTEX CALIBRATION STANDARD FOR OKLAHOMA METHODS 8020/8015 (MODIFIED)

NOTE: The following formulation is an example of a commercially available GRO/BTEX standard for the Oklahoma Methods 8020/8015 (modified). In this case, the formulation is that of Ultra UST-100. The components are in a methanol solution.

Component	Stock Standard Concentration (∝g/mL)	Intermediate Standard Concentration (∝g/mL)
1,2,4-Trimethylbenzene	1000	20
1,3,5-Trimethylbenzene	1000	20
Benzene	1000	20
Ethylbenzene	1000	20
Gasoline	10,000	200
m- & p-Xylenes	2000	20
Methyl t-Butyl Ether (MTBE)	1000	20
Naphthalene	1000	20
o-Xylene	1000	20
Toluene	1000	20

TABLE 4.

OKLAHOMA GRO METHOD CALIBRATION LEVELS

Calibration Levels (∝g/L)							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,2,4-Trimethylbenzene	0.5	2	5	10	20	50	100
1,3,5-Trimethylbenzene	0.5	2	5	10	20	50	100
Benzene	0.5	2	5	10	20	50	100

Ethylbenzene	0.5	2	5	10	20	50	100
m- & p-Xylene	1	4	10	20	40	100	200
Methyl t-Butyl Ether (MTBE)	0.5	2	5	10	20	50	100
Naphthalene	0.5	2	5	10	20	50	100
o-Xylene	0.5	2	5	10	20	50	100
Toluene	0.5	2	5	10	20	50	100
Total Xylenes	1.5	6	15	30	60	150	300
GRO - C_6-C_{10}	5.0	20	50	100	200	500	1000
Surrogate	0.75	3	7.5	15	30	75	150

TABLE 5.

TYPICAL COMMERCIAL GRO/BTEX SECOND-SOURCE STANDARD

NOTE: The following formulation is an example of a commercially available GRO/BTEX second-source standard. In this case, the formulation is that of Restek 30237. The components are in a methanol solution.

Component	Stock Standard Concentration (∝g/mL)	8015 GRO/NWTPH and AK101 Working Standard Concentration (∝g/mL)
Benzene	80	1.47
Ethylbenzene	94	1.72
Isopropylbenzene	12	0.22
m- & p-Xylenes	327	6.00
Methyl t-Butyl Ether (MTBE)	124	2.27
Naphthalene	25	0.46
o-Xylene	130	2.38
Toluene	399	7.32
Unleaded gasoline composite (compliant with Alaska Method AK101)	5500	100.8

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TABLE 6.

TYPICAL COMMERCIAL SECOND-SOURCE STANDARD FOR OKLAHOMA METHODS 8020/8015 (MODIFIED)

NOTE: The following formulation is an example of a commercially available GRO/BTEX second-source standard for the Oklahoma Methods 8020/8015 (modified). In this case, the formulation is that of Restek 300095. The components are in a methanol solution.		
Component	**Stock Standard Concentration (∝g/mL)**	**8015 GRO/NWTPH and AK101 Working Standard Concentration (∝g/mL)**
1,2,4-Trimethylbenzene	1000	20
1,3,5-Trimethylbenzene	1000	20
Benzene	1000	20
Ethylbenzene	1000	20
Gasoline	10,000	200
m- & p-Xylenes	2000	20
Methyl t-Butyl Ether (MTBE)	1000	20
Naphthalene	1000	20
o-Xylene	1000	20
Toluene	1000	20

TABLE 7.

TYPICAL RT STOCK STANDARDS

NOTE: The following formulations are examples of commercially available stock standards (in methanol)

Table 7A

TYPICAL RT REFERENCE STANDARD – Method 8015		
Component	**Stock Standard Concentration (∝g/mL)**	**Working Standard Concentration (∝g/mL)**
1,2,4-Trimethylbenzene (C_9)	1000	20
2,2,4-Trimethylpentane (C_8)	1500	30
2-Methylpentane (C_6)	1500	30
Benzene (C_6)	500	10
Ethylbenzene (C_8)	500	10
m-Xylene (C_8)	1000	20
Methyl t-Butyl Ether (MTBE)	2000	40
n-Heptane (C_7)	500	10
o-Xylene	1000	20
p-Xylene	1000	20
Toluene (C_7)	1500	30

Table 7B

Typical RT Marker Standard – AK101		
Component	**Stock Standard Concentration (ug/mL)**	**Working Standard Concentration (∝g/mL)**
n-Pentane (C_5)	2000	50
n-Hexane (C_6)	2000	50
n-Octane (C_8)	2000	50
n-Decane (C_{10})	2000	50
n-Dodecane (C_{12})	2000	50
n-Tridecane (C_{13})	2000	50

TABLE 8.
RECOMMENDED INSTRUMENT CONDITIONS

Purge-and-Trap Apparatus

Purge gas	Nitrogen or Helium
Purge gas flow rate	25-40 mL/min.
Purge temperature	35 ℃
Desorb temperature	185 ℃
Backflush inert gas flow	185 mL/min.
Desorb time	0.5 minutes
Purge time	11 minutes
Dry purge time	1 minutes

GC Conditions (Q)

Helium Column Pressure	20 psi
Initial Column Temperature	45 ºC for 2.00 minutes
First Temperature Ramp	9 ºC/minute & hold at 110 ºC for 0 minutes
Second Temperature Ramp	18 ºC/minute to 180 ºC
Third Temperature Ramp	30 ºC/minute to 250 ºC & hold for 4 minutes
FID Temperature	320 ºC

GC Conditions (S)

Helium Column Pressure	20 psi
Initial Column Temperature	45 ºC for 2.00 minutes
First Temperature Ramp	9 ºC/minute & hold at 110 ºC for 0.0 minutes
Second Temperature Ramp	18 ºC/minute to 180 ºC hold for 0.0 minutes
Third Temperature Ramp	230 ºC/minute to 250 ºC & hold for 2.00 minutes

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FID Temperature	320 ºC

TABLE 9.

PRESERVATION AND STANDARD PREP FOR MEDIUM LEVEL GRO SOILS

Container Received	TALS Prep Method	Analytical Method	Initial Volume	Final Volume	Holding Time	Notes
Unpreserved soil – glass jar/brass tube	5030B_H	8015 GRO	10 g	10 mL	14 days	--
EnCore™	5035A_M	8015 GRO/ OKGRO	5 g	5 mL	48 hours; 14 days	Preservation must be within 48 hours of sampling
Terra Core samples – 40 mL MeOH preserved VOA vial	5035FM_Calc	8015 GRO/ OKGRO	5 g	5 mL	14 days	MeOH not added by laboratory
AK101- 4 oz MeOH preserved jar	5035FM_MoistCor	AK101	25 g	25 mL	28 days	MeOH not added by laboratory; MeOH is surrogated in field at a concentration of 50ug/mL
Unpreserved soil – 40 mL VOA vial	5030B_H	NWTPH_Gx	5 g	10 mL	14 days	Extract good for 7 days

TABLE 10.

AMOUNT OF METHANOL EXTRACT NEEDED FOR ANALYSIS OF SOILS

GRO Approximate Concentration (∝g/g) [a]	Volume of Methanol Extract (∝L) [b]
0 – 1000	100
1000– 2000	50
2000 – 5000	10
≥ 5000	100 ∝L of 1/50 dilution [c]

Calculate the appropriate dilution factor for concentrations exceeding the concentrations in this table.

[a] This value may be based on a screening result or historical knowledge.

[b] The volume of methanol added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of methanol is necessary to maintain a volume of 100 ∝L for each blank, sample, and control.

[c] Dilute an aliquot of the methanol extract and then take 100 ∝L for analysis.

TABLE 11.

Alaska Method AK101 Quality Control Acceptance Criteria

Analyte	Spike Concentration		Control Limits	
	Water (mg/L)	Soil (mg/kg)	% Recovery	RPD (%)
GRO in Lab-Fortified Blanks[1]	0.1 – 1.0	5 - 100	60 - 120	20
Laboratory Sample[2] Surrogate Recovery (α,α,α-trifluorotoluene or bromofluorobenzene)	0.06	2.5	60 - 120	---
Field Sample Surrogate Recovery (α,α,α-trifluorotoluene or bromofluorobenzene)	0.05	2.5	50 - 150	---
Continuing Calibration	1.0	---	75 - 125	---
Calibration Verification	1.0	---	75 - 125	---

[1] This is the same as the TAL Denver laboratory control sample (LCS).

[2] Laboratory samples are control samples that are spiked in the laboratory, i.e., LCS, method blank, MS, and MSD.

The quality control criteria listed in this table represent the minimum acceptable levels, using highly organic soil matrices. Higher performance may be required on some projects.

TABLE 12.

Oklahoma GRO Analyte/Peak Number

Analyte	Peak Number
Methyl tert-butyl ether	1
Benzene	2
α,α,α-Trifluorotoluene	3
Toluene	4
1-Chloro-4-fluorobenzene	5
Chlorobenzene	6
Ethylbenzene	7
m-Xylene & p-Xylene	8
o-Xylene	9
1,3-Dichlorobenzene	11
1,4-Dichlorobenzene	12
1,2-Dichlorobenzene	13
Naphthalene	14

TABLE 13.

Method 8015 and NWTPH-Gx Instrument QC and Sample Spiking Amounts

QC Type	Matrix	Sample Amt	Spiking Soln ID	Volume Spiked	On-Column Conc (x)	Conc (µg/L or mg/Kg)
RTC	Water or Soil	50 mL	8015ISscs	50 µL	--	--
			8021 GRO-CSA	50 µL	--	--
CCVRT or CCV	Water or Soil	50 mL	8015ISscs	50 µL	60	60
			8015GRO-CSA	50µL	200	200
MB	Water	5 mL	8015ISscs	50 µL	60	60
	Soil	5 g	8015GROTFTStk	50 µL	60	3
LCS/MS/MSD	Water	5 mL	8015GRODCS	5 µL	100	100
			8015ISscs	5 µL	60	60
	Soil	5 g	8015GRODCSStk	5 µL	100	4.5
			8015GROTFTStk	5 µL	60	3
Sample Surrogate	Water	5 mL	8015ISscs	5 µL	60	60
	Soil	5 g	8015GROTFTStk	5 µL	60	3

TABLE 14.
Method AK101 Instrument QC and Sample Spiking Amounts

QC Type	Matrix	Sample Amt	Spiking Soln ID	Volume Spiked	On-column Conc	Conc (µg/L or mg/Kg)
CCVRT or CCV	Water or Soil	5 mL	AK101Surrogate*	5µL	60	60
			AKGROCSA	50µL	200	200
MB	Water	5 mL	AK101Surrogate	5µL	60	60
	Soil	5 g	8021GROTFTStk	5µL	60	3
			AK101-BFB	5µL	60	3
LCS	Water	5 mL	8015GRODCS	50µL	100	100
			AK101Surrogate	5µL	60	60
	Soil	5 g	8015GRODCSStk	5µL	100	4.5
			8021GROTFTStk	5µL	60	3
MS/MSD	Water	5 mL	8015GRODCS	50µL	100	100
			AK101Surrogate	5µL	60	60
	Soil	5 g	8015GRODCS	5µL	100	4.5
			AK101-BFB	5µL	60	3
Sample Surrogate	Water	5 mL	AK101Surrogate	50µL	60	60
	Soil	25 g	8021GROTFTStk	5 µL	60	3

*All surrogates added by instrument at time of purge

TABLE 15.
Method Okla_GRO Instrument QC and Sample Spiking Amounts

QC Type	Matrix	Sample Amt	Spiking Soln ID	Volume Spiked	On-column Conc (x)	Conc (µg/L or mg/Kg)
CCV or CCV	Water or Soil	5 mL	8021-IS/SCSmo	5 µL	60	60
			OKGRO CSA	5 µL	200	200
MB	Water	5 mL	8021-IS/SCS	5 µL	60	60
	Soil	5 g/5mL	8021GROTFTStk	5 µL	60	2
LCS/MS/MSD	Water	5 mL	8021GRO SS/DCS	5 µL	200	200
			8015ISscsSCS	5 µL	60	60
	Soil	5 g/5mL	OKGRO SS Stk	5 µL	200	10
			8021GROTFTStk	5 µL	60	2
LLCS	Water	5 mL	OKGRO_ICALmix	5 µL	40	40
Sample Surrogate	Water	5 mL	8015ISscsSCS	5 µL	60	60
	Soil	5 g/5mL	8021GROTFTStk	5 µL	60	2

*All surrogates added by instrument at time of purge

APPENDIX A –

Sample Collection Instructions for Alaska Method AK101 GRO/BTEX

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TestAmerica Denver
Volatile (AK101 GRO/BTEX) Sample Collection Instructions

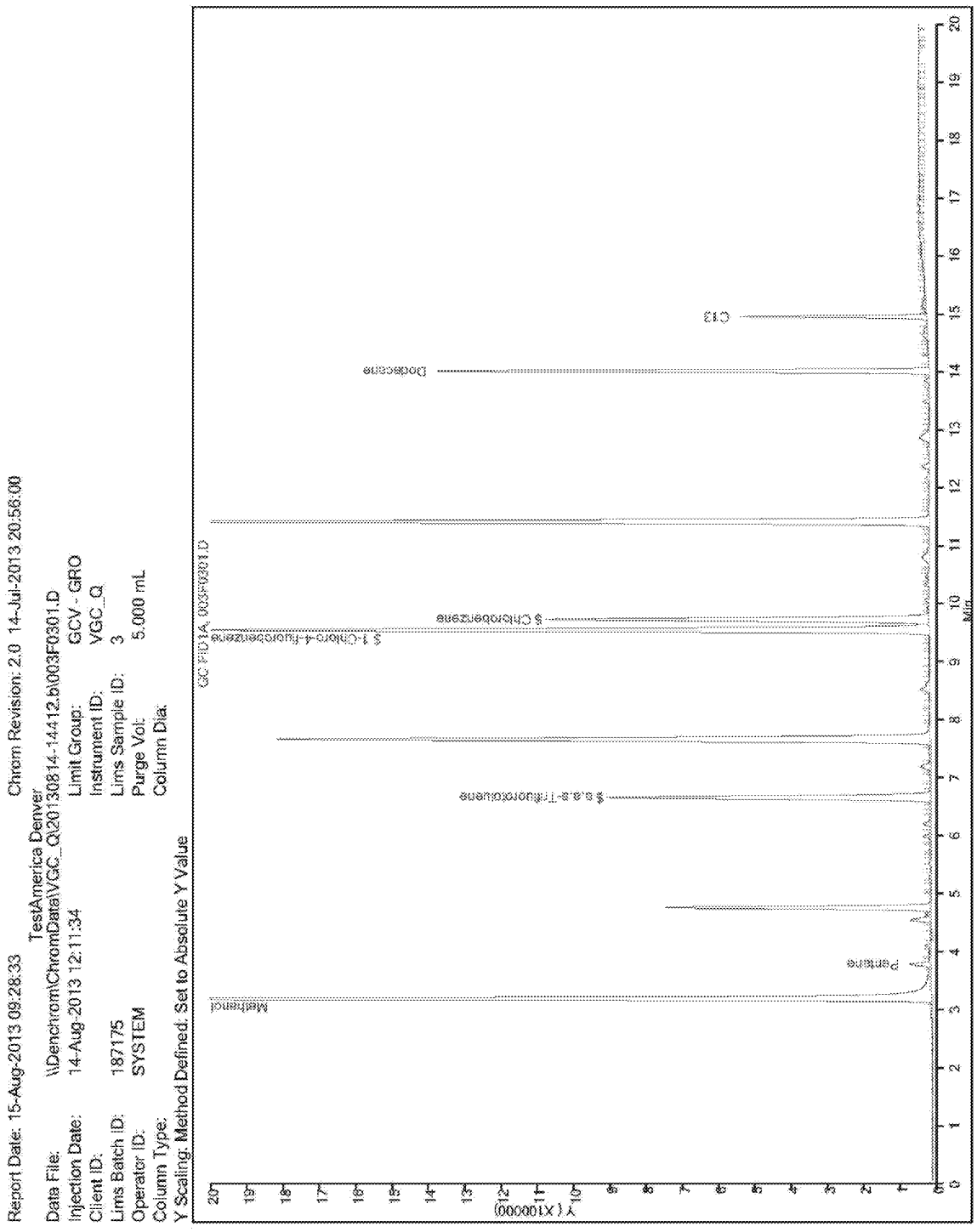
Soil Samples:

1. Soil samples should be collected in 4-oz amber jars that are pre-tared. Using a pre-tared jar ensures that the lab can very accurately determine the sample mass. TestAmerica Denver provides these containers. Client should not place any additional labels/tape on the jars or switch lids between jars.
2. The sample should be collected with minimum disturbance.
3. **The recommended amount of sample to collect is 25 to 50g, which is approximately 1 inch of soil inside the jar**.
4. The vial containing 25-mL of methanol/surrogate solution (also provided by TestAmerica Denver) is then carefully poured into the jar.
5. **The soil sample MUST be completely submersed in the methanol/surrogate solution**. If too much sample is collected and therefore the sample is not completely submersed in the methanol/surrogate solution, sample integrity has been compromised and the lab will issue the final report with a qualifier for the sample. If additional MeOH has been added or MeOH was lost for any reason the laboratory must be notified.
6. Samples must be refrigerated or iced to 0-6℃.
7. A trip blank (provided by the lab) must accompany each shipping container and should be stored with the field samples.
8. An additional sample of the same soil to be analyzed for GRO should be collected into a 4-oz unpreserved amber jar for percent moisture analysis in order to report results on a dry weight basis.

Aqueous Samples:

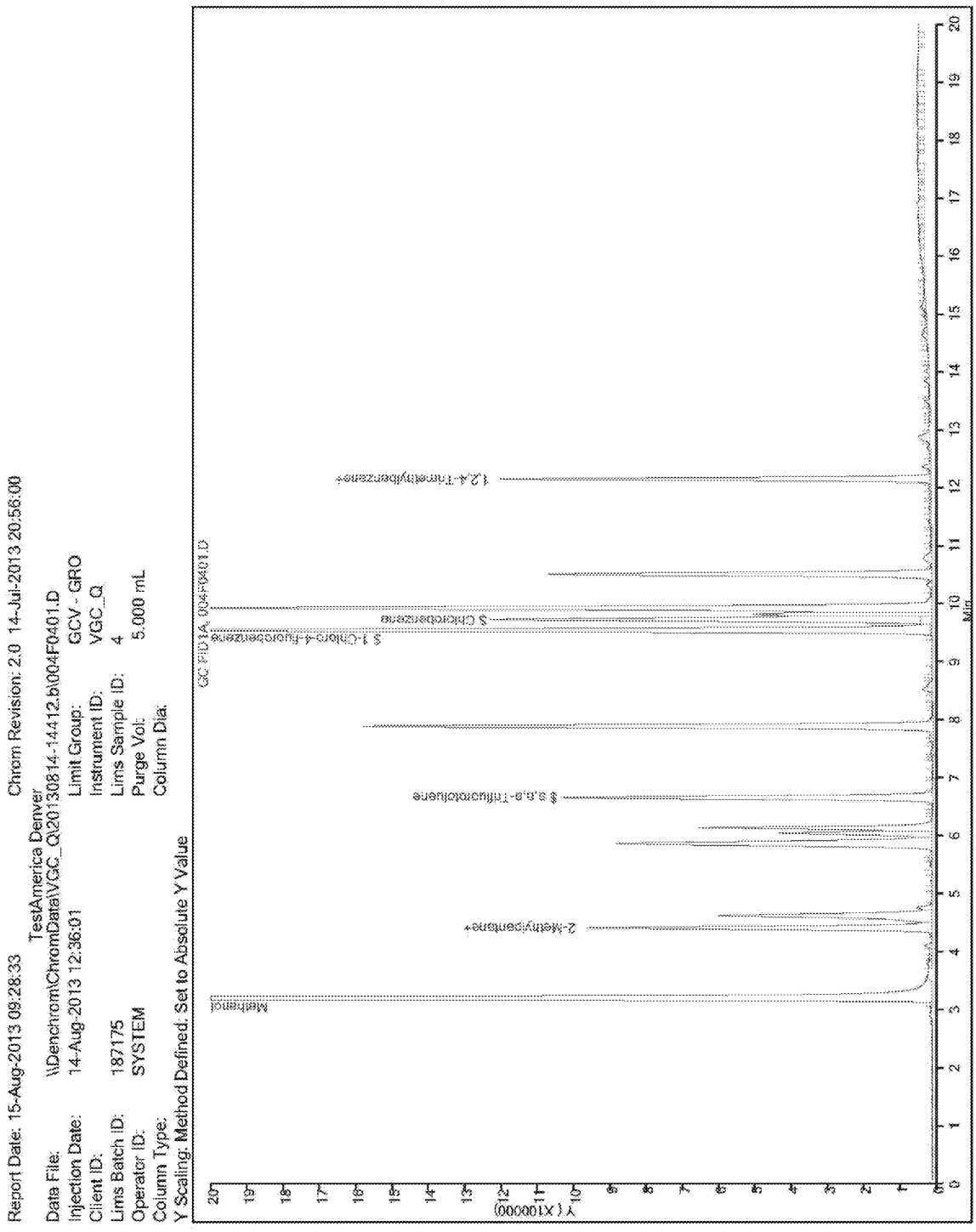
1. Aqueous samples should be collected in 40-mL HCl preserved glass VOA vials. TestAmerica Denver provides these containers. Three vials per sample are required.
2. Samples should be collected without agitation and **WITHOUT HEADSPACE**.
3. Samples must be refrigerated or iced to 0-6℃.
4. An aqueous trip blank (provided by the lab) must accompany each shipping container and should be stored with the field samples.

Figure 1
Sample Chromatogram for Alkane RT Marker Standard



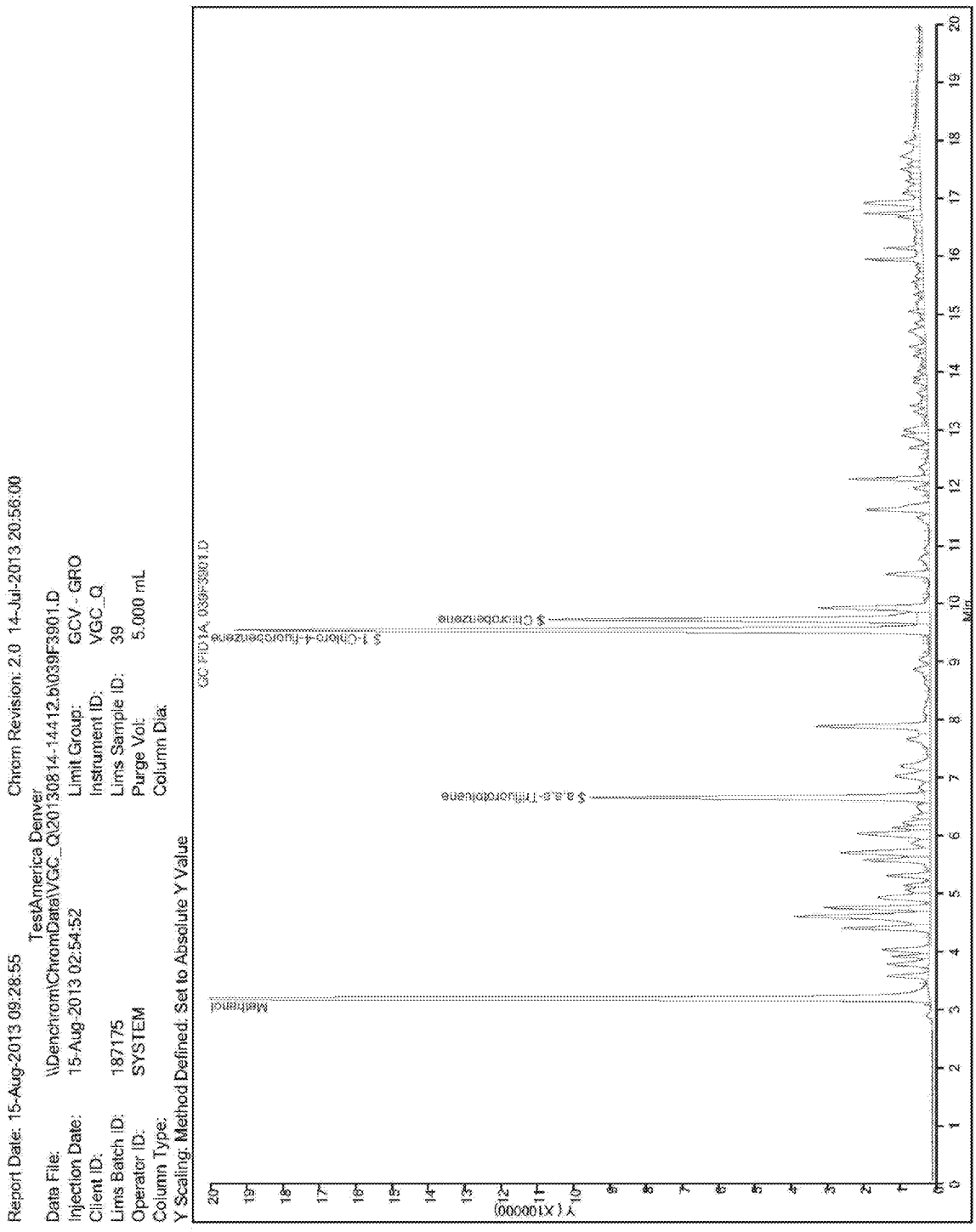
Note: Actual retention times will vary by instrument and column.

Figure 2
Sample Chromatogram for GRO Retention time Standard



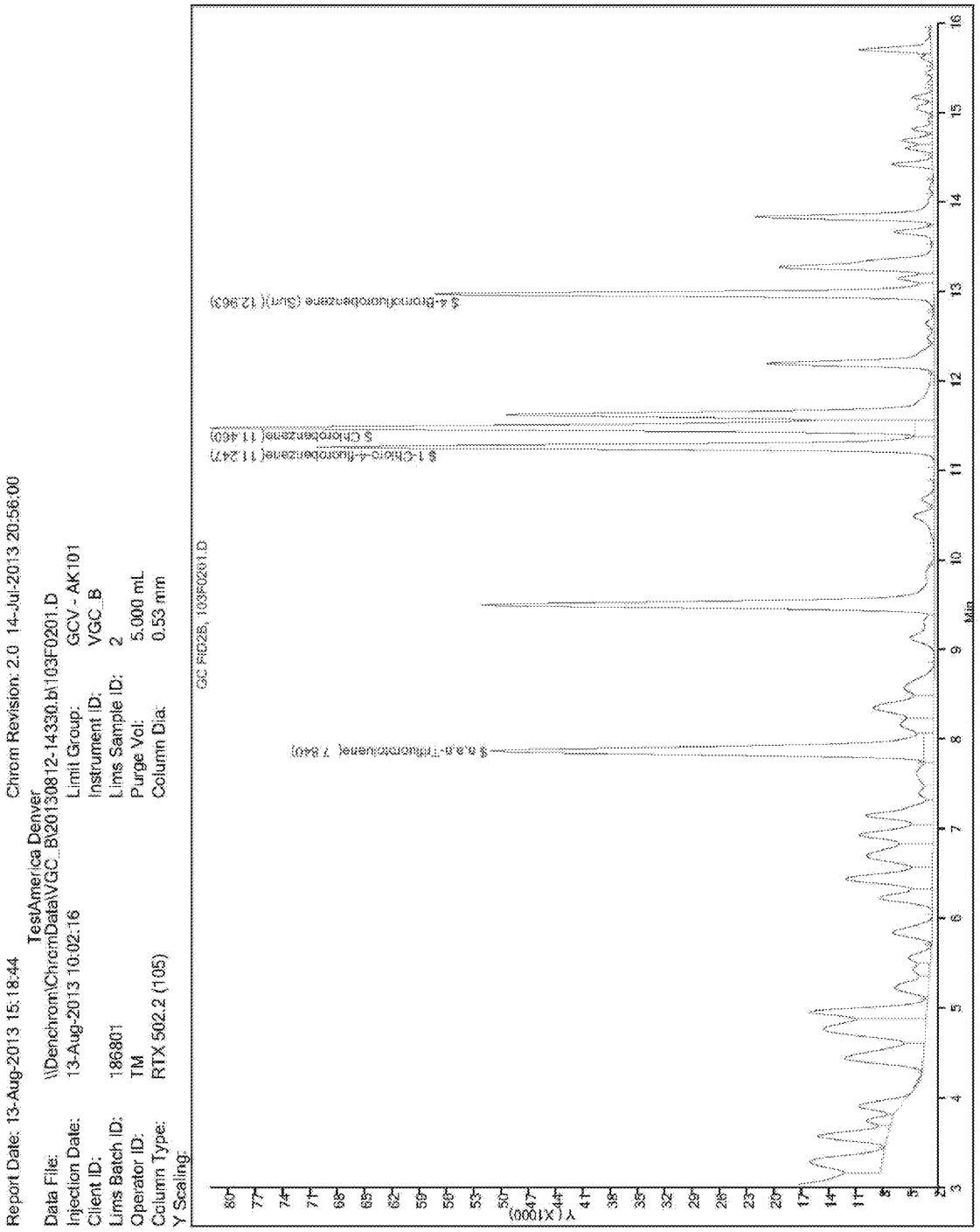
Note: Actual retention times will vary by instrument and column.

Figure 3
Sample Chromatogram for 8015 GRO CCV



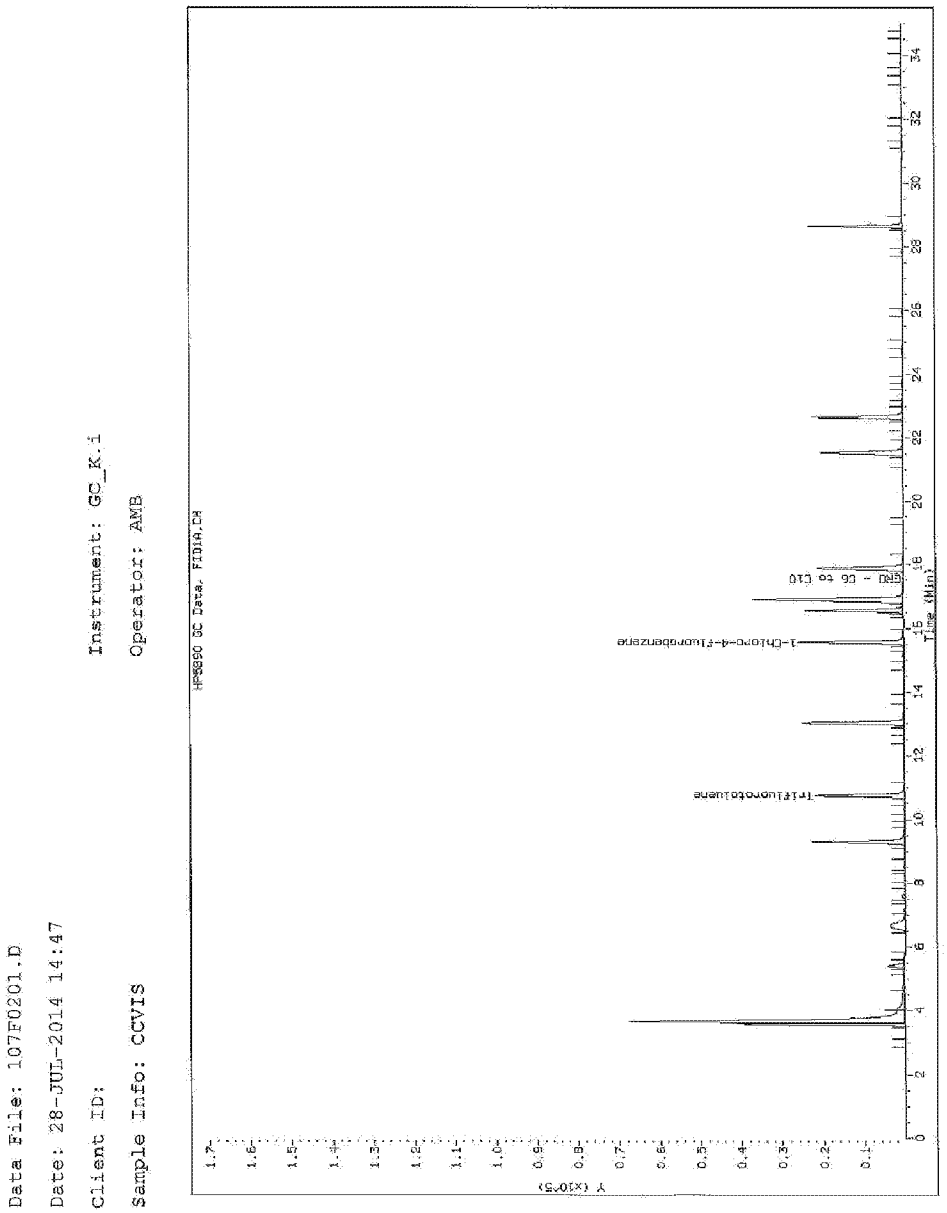
Note: Actual retention times will vary by instrument and column.

Figure 4
Sample Chromatogram for AK101 CCV



Note: Actual retention times will vary by instrument and column.

Figure 6
Sample Chromatogram for OKGRO CCV on FID



Note: Actual retention times will vary by instrument and column.

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Title: Chlorinated Pesticides [EPA 8081A & 8081B]

Approvals (Signature/Date):

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Heather Fiedler Technical Manager	Date	Reed Pottruff Health & Safety Manager / Coordinator	Date
9/19/19	9/19/19
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1.0 Scope and Application

1.1 This standard operating procedure (SOP) describes the determination of chlorinated pesticides using the methodology described in EPA SW-846 Method 8081A and 8081B with 8000B, 8000C or 8000D as specified by project requirements.

1.2 This SOP is applicable to the gas chromatographic (GC) analysis of extracts of soil and water samples. Table 1 lists the compounds that can be determined by this method and their associated routine reporting limits (RLs).

1.3 This SOP does not include the procedures for extracting soil and water samples. Refer to the following SOPs for sample extraction procedures:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW-846 3510C
DV-OP-0007	Concentration of Organic Extracts, SW-846 3510C, 3520C, 3540C, and 3550C
DV-OP-0016	Ultrasonic Extraction of Solid Samples by SW-846 3550C
DV-OP-0015	Microwave Extraction of Solid Samples by SW-846 3546

1.4 Analytes, Matrix(s), and Reporting Limits

See Table 1 for analytes and reporting limits by matrix.

1.5 This SOP contains a Large Volume Injection (LVI) procedure. This procedure has not been approved by the State of South Carolina and therefore no samples from South Carolina may be analyzed using LVI.

2.0 Summary of Method

2.1 Sample Preparation

2.1.1 Chlorinated pesticides are extracted from a one-liter water sample with methylene chloride using a separatory funnel (Method 3510C). Detailed instructions are given in SOP DV-OP-0006. The methylene chloride extract is exchanged to hexane as described in SOP DV-OP-0007. An alternate procedure has been developed using a lower volume of sample (250 mL to a final volume of 5 mL) and a larger injection volume in order to minimize shipping requirements and conserve the reagents needed for extraction.

2.1.2 Chlorinated pesticides are extracted from a 30-gram soil subsample into a 50:50 acetone-methylene chloride solution by sonication (Method 3550C) or by microwave extraction (Method 3546). The extract is dried and exchanged to hexane. Detailed instructions are given in SOPs DV-OP-0016 and DV-OP-0015.

2.1.3 SOP DV-OP-0007 provides instructions for the concentration and cleanup

of sample extracts. Florisil is used to clean extracts that show color or when requested in order to minimize interferences when they are observed in the analysis. Sulfur is removed if observed. All extracts are in hexane and the final extract volume is 10 mL. For the LVI method the final extract volume is 5 mL.

2.2 Analysis

2.2.1 Samples are analyzed using a gas chromatograph equipped with dual columns and dual electron capture detectors (ECDs).

2.2.2 The instrument is calibrated using internal standards. Compounds are identified by their retention time on the columns.

2.2.3 Positive results from the primary column are confirmed with a second, dissimilar column. The laboratory maintains a total of four dissimilar columns for additional confirmation capability.

3.0 Definitions

3.1 Single-Component Pesticides: A pesticide formulation that consists of a single chemical compound. Most of the analytes determined by this procedure are single-compound pesticides.

3.2 Multi-Component Pesticides: A pesticide formulation that consists of more than one chemical compound. Toxaphene and Technical Chlordane are production mixtures of multiple compounds. Toxaphene is manufactured by the chlorination of camphenes, which produces a variety of compounds, not all of which are chromatographically resolved. Technical Chlordane is produced by the chlorination of a mixture of camphenes and pinenes.

3.3 Chlordane: As just described, Technical Chlordane (CAS# 12789-03-6) is a mixture of compounds. Method 8081A, Section 7.6.2 and Method 8081B, Section 11.6.2 note that Technical Chlordane includes at least 11 major components and 30 minor components, and adds "the exact percentage of each [*cis*-chlordane and *trans*-chlordane] in the technical material is not completely defined, and is not consistent from batch to batch." The laboratory has found that manufacturing lots of Technical Chlordane produced at different times or at different production facilities have different ratios of the key components. For this reason, it is more common to analyze for the major components of technical Chlordane (α-Chlordane, γ-Chlordane, and heptachlor) instead of analyzing for the total mixture. For the purpose of reporting results under this SOP, the following compounds are reported. Alpha-chlordane (*cis*-chlordane) CAS # 5103-71-9 and gamma-chlordane (*trans*-chlordane) CAS # 5103-74-2. trans-Chlordane has also been identified as beta-chlordane. The laboratory may also report chlordane (not otherwise specified) or, n.o.s under CAS# 57-74-9.

3.4 The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and SOP DV-QA-003P.

3.5 Lower Limit of Quantitation (LLOQ): The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be > the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

4.0 Interferences

4.1 Contamination by carryover can occur when a low concentration sample is analyzed immediately following a high concentration sample. It is the laboratory's policy to reanalyze any samples that follow an unusually concentrated sample (well above the high level calibration standard) and that show detectable levels of the same compounds that appeared in the preceding concentrated sample.

4.2 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector. Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.3 Sulfur will interfere, and, when observed, is removed using cleanup procedures described in SOP DV-OP-0007. An NCM which indicates the lot number of the materials used for cleanup is provided whenever a cleanup procedure is used.

4.4 Soil and water sample extracts are subject to Florisil cleanup when the extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Florisil removes low- to medium-molecular weight polar organic interferences from sample extracts. One limitation for this cleanup method is that recoveries for the most polar compounds, endosulfan sulfate and endrin aldehyde in particular, will be lower. Florisil has been observed to remove the compound kepone and is not used where the determination of kepone is required. Instructions for performing Florisil cleanups can be found in SOP DV-OP-0007. An NCM which indicates the lot number of the materials used for cleanup is provided whenever a cleanup procedure is used.

5.0 Safety

Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

5.1.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.1.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.1.4 The ECD contains a ^{63}Ni radioactive source. All ^{63}Ni sources shall be leak tested every six months, or in accordance with the facility's radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the Radiation Safety Officer shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment. Follow the proper procedures and precautions for the safe handling of radioactive materials when handling the ECDs in the event that leakage may have occurred.

5.1.5 As a safety precaution, all standards, samples, and extracts are handled in an approved fume hood.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure

Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects are exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 An analytical system complete with a gas chromatograph and dual ECD (Ni-63) detectors is required. A data system capable of measuring peak area and/or height is required. The instruments typically used for this method are HP 6890 instrument C and HP 6890N for instruments P1 and P2.

6.2 An analytical balance capable of weighing to 0.01 g.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.4 Columns

6.4.1 Primary Column: CLPI, 30 m X 0.32 mm id (used in instruments P1 and P2).

6.4.2 Secondary Column: CLPII, 30 m X 0.32 mm id (used in instruments P1 and P2).

6.4.3 Additional columns that can be used for confirmation include 30 m X 0.32 mm id RxiSil 35-MS or Rxi-XLB (used in instrument C).

6.5 Autosampler vials, crimp-top cap with PTFE-faced septa

6.6 Siltek Y-splitter, thermogreen septa, Siltek guard columns, ferrules, deactivated injection port liners (Agilent Ultra Inert, Siltek, or Sky liners all work well), Siltek glass wool, gold plated seals.

6.7 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.8 Class A volumetric flasks various sizes.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.1 Reagents

7.1.1 Hexane, pesticide grade; each lot tested for purity prior to use per SOP CA-Q-S-001.

7.1.2 Carrier gas, ≥ 99.99999% pure hydrogen or helium

7.1.3 Make-up gas, ≥ 99.99980% pure nitrogen

7.2 Standards Verification

All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.3 Storage of Stock Standards

7.3.1 Standards are purchased from commercial vendors and are received as certified solutions in flame sealed ampoules. Neat stocks with applicable certification may also be used. Stock standards are stored refrigerated at ≤ 6 °C. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before using.

7.3.2 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced at least every six months or sooner if comparison with check standards indicates a problem. Kepone in particular may demonstrate signs of degradation faster than the other compounds, and/or the expiration date. Endosulfan I and II appear to degrade in the presence of methanol. gamma-BHC appears to degrade in the presence of acetone.

7.4 Calibration Stock Standards

NOTE: The availability of the specific commercial standard solutions upon which the following sections are based may change at any time. As a result, it may be necessary to alter the dilution scheme presented herein to accommodate changes in stock standard concentrations. All such changes are documented in the standards preparation records.

7.4.1 Routine Pesticide AB Mix Stock Standard, 2,000 µg/mL

The routine pesticide AB mix stock standard (**8081ABResPS**), Restek 32415, contains all of the "routine" single-component pesticides, as identified in Table 1 with the addition of Hexachlorobenzene at 100 µg/mL (**8081HCBStkPS**) (Accustandard APP-9-112), Mirex at 100 µg/mL (**8081MirxStkPS**)(Accustandard P-066S) and Isodrin at 1000 µg/mL (**8081IsodrinPS**) (Accustandard P471S-10x).

7.4.2 Surrogate B Mix Stock Standard, 200 µg/mL

The surrogate B mix stock standard (**AR_SURR_RES**) (Restek 32000) contains decachlorobiphenyl (DCB) and tetrachloro-*m*-xylene (TCMX).

7.4.3 Toxaphene Stock, 5000 µg/mL

The Toxaphene stock standard (**8081ToxPS**) (Restek 32071) contains a specific production mixture of Toxaphene. This mixture does not necessarily match all possible production mixtures that could be found in the environment. This can present problems for Toxaphene quantitation (see Section 12).

7.4.4 Chlordane Stock, 5000 µg/mL

The Chlordane stock (**8081ChlrStkPS**) (Restek 32072) contains Technical Chlordane (CAS# 12789-03-6).

7.4.5 Appendix IX Calibration Stock

The Appendix IX stock calibration mixture (**8081AP9StkPS**) (Accustandard S-6880 custom) contains the compounds at the concentrations listed in the following table. Propachlor at 1000 ug/mL is also added to the mixture (Accustandard P-215S-10x).

Appendix IX Calibration Stock Standard

Compound	Concentration (µg/mL)
2,4'-DDD	100
2,4'-DDE	100
2,4'-DDT	100
Chlorobenzilate	1,000
Chlorpyrifos	500
Diallate	10,000
Dicofol	1,000
Propachlor	1,000
Kepone	1,000
DBPP	5,000

7.4.6 Internal Standard stock

A commercially prepared stock standard solution is obtained that contains the internal standard 1-bromo-2-nitrobenzene in acetone, at a concentration of 1000 µg/mL. The current vendor is RESTEK catalog #32279, other vendors may be used.

7.4.7 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.4.7.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in Section 7.2 above.

7.4.7.2 Non-routine standards will be stored and treated as described in Section 7.3 above or as specified by the manufacturer.

7.4.7.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.4.7.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.4.7.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.5 Intermediate Level Calibration Standards

7.5.1 Routine Pesticide Mix C Intermediate Calibration Standard, 1.0 µg/mL (**8081ABCalStk**). The intermediate level calibration standard for routine pesticide compounds including Hexachlorobenzene and Mirex is prepared by diluting the AB (Section 7.4.1) and B (Section 7.4.2) mix stock standards in hexane to 100 mL final volume as follows (all compounds are the same final concentration):

Mix C Intermediate Calibration Standard

Stock AB mix (mL)	Stock B (mL)	Mirex & HCB (mL)	Isodrin (mL)	Final Concentration of Each Pesticide (µg/mL)
0.05	0.5	1.0	0.1	1.0

7.5.2 Appendix IX Intermediate Calibration Standard

The Appendix IX intermediate level calibration standard (**8081AP9CalStk**) is prepared by diluting 0.5 mL of the Appendix IX stock standard (Section

7.4.5) and 0.5 mL of propachlor stock with hexane to a final volume of 50 mL, which results in the following concentrations:

Appendix IX Intermediate Calibration Standard

Compound	Concentration (µg/mL)
2,4'-DDD	1.0
2,4'-DDE	1.0
2,4'-DDT	1.0
Chlorobenzilate	10.
Chlorpyrifos	5.0
DBPP	50.
Diallate	100.
Propachlor	10.
Dicofol	10.
Kepone	10.

7.6 Working Level Calibration Standards

7.6.1 Routine Pesticide AB Mix Working Level Calibration Standards

The following volumes of the 1.0 µg/mL Mix C intermediate standard (Section 7.5.1) are diluted to 100 mL with hexane to produce calibration standards at 6 concentration levels, as summarized in the following table:

AB Mix Working Level Calibration Standards

Level	Volume of Mix C Intermediate Std (mL)	Final Concentration (µg/mL)
1 (**8081IcalL1**)	0.4	0.0040
2 (**8081IcalL2**)	1.0	0.010
3 (**8081IcalL3**)	2.5	0.025
4* (**8081IcalL4**)	5.0	0.050
5 (**8081IcalL5**)	7.5	0.075
6 (**8081IcalL6**)	10	0.10

* *This level is used as the Continuing Calibration Verification (CCV) standard. As a result, it may be convenient to make a larger volume of this calibration level, by diluting 12.5 mL of the intermediate standard with hexane to a final volume of 250 mL.*

7.6.2 Toxaphene Working Level Calibration Standards

The following volumes of the 5000 µg/mL Toxaphene stock standard (Section 7.4.3) are diluted with hexane to the final volumes indicated in the following table:

Toxaphene Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1 (**8081ToxL1**)	0.004	100	0.20
2 (**8081ToxL2**)	0.01	100	0.50
3 (**8081ToxL3**)	0.02	100	1.0
4 (**8081ToxCCVL4**)	0.1	250	2.0
5 (**8081ToxL5**)	0.1	100	5.0
6 (**8081ToxL6**)	0.2	100	10.0
Level 4 is used as the CCV standard when running a 5 pt curve.			

7.6.3 Chlordane Working Level Calibration Standards

A chlordane substock (**8081ChlrWSPS**) is prepared by diluting 0.200 mL of the stock described in section 7.4.4 to a final volume of 10 mL with hexane. The following volumes of the resulting 100 µg/mL Chlordane working stock standard are diluted with hexane to the final volume indicated in the following table:

Chlordane Working Level Calibration Standards

Level	Volume of Stock Std (mL)	Final Volume (mL)	Final Concentration (µg/mL)
1 (**8081ChlorL1**)	0.05	100.0	0.05
2 (**8081ChlorL2**)	0.2	100.0	0.20
3 (**8081ChlorL3**)	0.5	100.0	0.50
4* (**8081ChlorL4**)	1.0	100.0	1.0
5 (**8081ChlorL5**)	2.0	100.0	2.0
* *This level is used as the CCV standard.*			

7.6.4 Appendix IX Working Level Calibration Standards

The following volumes of the Appendix IX intermediate calibration standard (Section 7.5.2) are diluted with hexane to a final volume of 1.0 mL. The following table summarizes the final compound concentration ranges for each calibration level. The concentration for each compound at each level is given in Table 3.

Appendix IX Working Level Calibration Standards

Level	Volume of Intermediate Std (mL)	Final Compound Concentration Range (µg/mL)

1	Dilute 1ml Level 2 to 5 ml	0.001 - 0.10
2	0.005	0.005 – 0.50
3	0.010	0.01 - 1.0
4	0.025	0.025 - 2.5
5*	0.035	0.035 - 3.5
6	0.050	0.05 - 5.0
7	0.100	0.1 - 10
* *This level is used as the CCV. Because some compounds in this standard are not stable, it is not recommended to make extra volume of the level 5 standard.*		

7.7 Working Level Calibration Standards for the large volume injection (LVI) procedure

The standards for the LVI method can be prepared using the associated full volume standards described in the previous section 7.6 by mixing equal parts of standard and reagent grade hexane (a 2x dilution) or by simply substituting the appropriate standard from section 7.6 for the corresponding LVI standard that is at the same concentration. Likewise, the LVI standards can be prepared from stock materials. In any case the method of preparation will be completely documented in the standards preparation records. The tables below indicate a typical preparation protocol.

7.7.1 Routine Pesticide AB Mix Working Level LVI Calibration Standards

Calibration standards are prepared by diluting equal volumes of the corresponding calibration standard level from section 7.6.1 with hexane. Calibration standard Level 1 from section 7.6.1 is used to prepare calibration standard Level 1 for the LVI, etc.

AB Mix Working Level LVI Calibration Standards

Level	Volume of Corresponding Std Level (mL) from section 7.6.1 to 2mL final vol.	Final Concentration (µg/mL)
1	1.0	0.002
2	1.0	0.005
3	1.0	0.0125
4*	Use level 3 section 7.6.1	0.025
Level	**Volume of Corresponding Std Level (mL) from section 7.6.1 to 2mL final vol.**	**Final Concentration (µg/mL)**
5	1.0	0.0375
6	Use level 4 section 7.6.1	0.05
* *This level is used as the Continuing Calibration Verification (CCV) standard.*		

7.7.2 Toxaphene Working Level Calibration Standards

Calibration standards for LVI are prepared by diluting equal volumes of the corresponding standard level from section 7.6.2 with reagent hexane.

Toxaphene Working Level Calibration Standards

Level	Volume of Corresponding Std Level (mL) from section 7.6.2	Final Volume (mL)	Final Concentration (µg/mL)
1	1.0	2.0	0.1
2	1.0	2.0	0.25
3	Use Level 2	-	0.5
4	Use Level 3	-	1.0
5	1.0	2.0	2.5
6	Use Level 5	-	5.0
• *Level 4 is used as the CCV standard when running a 5 pt curve.*			

7.7.3 Chlordane Working Level LVI Calibration Standards

Calibration standards for LVI are prepared by diluting equal volumes of the corresponding standard level from section 7.6.3 with reagent hexane.

Chlordane Working Level LVI Calibration Standards

Level	Volume of Corresponding Std Level (mL) from section 7.6.3	Final Volume (mL)	Final Concentration (µg/mL)
1	1.0	2.0	0.025
2	1.0	2.0	0.1
3	1.0	2.0	0.250
4*	Use Level 3	-	0.5
5	Use Level 4	-	1.0
6	Use Level 5	-	2.0
* *This level is used as the CCV standard.*			

7.7.4 Appendix IX Working Level LVI Calibration Standards

Calibration standards for LVI are prepared in the same manner as for the dilution scheme presented in section 7.6.4 by using a 2x dilution of the Appendix IX intermediate calibration standard from section 7.5.2.

Appendix IX Working Level LVI Calibration Standards

Level	Volume of 2x dilution of Intermediate Std (mL)	Final Compound Concentration Range (µg/mL)
1	Dilute 1 ml of level 2 to	0.0005 - 0.05

	5 ml	
2	0.005	0.0025 – 0.25
3	0.010	0.005 – 0.5
4*	0.025	0.0125 – 1.25
5	0.035	0.0175 – 0.1.75
6	0.050	0.025- 2.5
7	0.100	0.05 - 5
* *This level is used as the CCV. Because some compounds in this standard are not stable, it is not recommended to make extra volume of the level 4 standard.*		

7.8 Second-Source Standards for Initial Calibration Verification (ICV)

The second-source stock standards are purchased from a vendor as different from the one that supplied the stock calibration standards.

7.8.1 Routine Pesticide AB Mix ICV Stock Standard, 2,000 µg/mL, (with Mirex at 100 µg/mL, Isodrin at 5000 ug/mL, HCB at 1000 ug/mL)

Commercial standards containing all single-component pesticide compounds are obtained from a vendor different from the one that supplied the calibration stock standard. The AB mix is prepared from a standard supplied from Restek (**8081ABResSS**) as a separate second source preparation cat # 32415.sec. Typically, the standards are obtained from Ultra Scientific standard EPA-1125 for Hexachlorobenzene (**8081HCBStkSS**), standard PST-720S for Mirex (**8081MirxStkSS**), and standard EPA-1131 for Isodrin (**8081IsodrinSS**).

The current toxaphene second source (**8081ToxSS**) is AccuStandard P-093S-H-10X and it is prepared by diluting 5 µL of the stock standard to 5 mL with hexane.

The current chlordane second source (**8081ChlrStkSS**) is prepared by Restek as a separate second source preparation cat# 32072.sec at a concentration of 5,000 µg/mL. A working substock (**8081ChlSSL3**) is prepared by diluting 0.2 mL of the stock to a final volume of 10 mL with hexane and the working standard is prepared by diluting 5 µL of the working substock standard to 10 mL with hexane.

7.8.2 Appendix IX ICV Stock Standard (8081AP9StkSS)

Commercial standards are obtained at the same concentrations as shown for the calibration stock standards in Section 7.4.5, but from a different vendor (typically Ultra Scientific standard CUS-14331). A second source for propachlor from Ultra PST-865M100A01 at 100 µg/mL is also added to this stock (**8081PropachSS**).

Compound	Concentration (µg/mL)
2,4'-DDD	10
2,4'-DDE	10
2,4'-DDT	10
Chlorobenzilate	100
Chlorpyrifos	50
DBPP	5,000
Diallate	1,000
Propachlor	100
Dicofol	100
Kepone	100

7.8.3 Surrogate ICV Stock Standards, 200 µg/mL

Commercial standards (typically Ultra Scientific standard ISM-320) are obtained containing decachlorobiphenyl (DCBP) and tetrachloro-*m*-xylene (TCMX).

7.8.4 ICV Intermediate Level Standards, 1.0 µg/mL

The ICV intermediate level calibration standard for routine pesticide compounds (**8081ABICVStk**) is prepared by diluting the AB, Hexachlorobenzene, and Mirex, and surrogate stock standards (Sections 7.8.1) with hexane to a final volume of 25 mL as summarized in the table below. All compounds in the intermediate standard are at the same final concentration, i.e., 1.0 µg/mL.

Second-Source ICV Intermediate Standard

Vol of AB (mL)	Vol of Mirex Stock (mL)	Vol of Isodrin (mL)	Vol of Surrogate Stock (mL)	Vol of HCB (mL)	Final Conc (µg/mL)
0.0125	0.25	0.005	0.125	0.025	1.0

7.8.5 Routine Pesticide ICV Working Level Standard, 0.025 µg/mL (8081ICVL3)

The working level ICV standard for the routine pesticide compounds is prepared by diluting the ICV intermediate standard (Section 7.8.4) in hexane follows:

Routine Pesticide Second-Source ICV Working Level Standard

Volume of Intermediate Standard (mL)	Final Volume (mL)	Final Concentration (µg/mL)
2.5	100	0.025

7.8.6 Appendix IX ICV Working Level Standard

The working level ICV standard for the Appendix IX compounds is prepared by diluting 0.0025 mL of the second-source Appendix IX stock standard (Section 7.8.2) and 0.0025 mL of the propachlor stock with hexane to a final volume of 1 mL. The following table lists the final concentration of each pesticide:

Appendix IX ICV Working Level Standard

Pesticide	Final Concentration (µg/mL)
2,4'-DDD	0.025
2,4'-DDE	0.025
2,4'-DDT	0.025

Pesticide	Final Concentration (µg/mL)
Chlorobenzilate	0.25
Chlorpyrifos	0.125
Diallate	2.5
Propachlor	0.25
Dicofol	0.25
Kepone	0.25

Note: The LVI method ICV can be prepared from the corresponding ICV from above by mixing equal parts of the ICV above with reagent hexane (a 2x dilution).

7.9 Continuing Calibration Verification (CCV) Standards

The level 4 AB mix working calibration standard (Section 7.6.1) and the level 4 Appendix IX working calibration standard (Section 7.6.4) are used as the CCV standards.

7.10 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL standard.

7.11 Laboratory Control Standard (LCS) Spike Solution, 0.5 µg/mL

The LCS working spike stock solution is prepared by diluting 0.25 mL of the AB mix stock standard Restek 32415 (2000 ug/mL) in acetone (see Section 7.4.1) to a final volume of 10 mL in a volumetric flask. The LCS spike solution is prepared fresh each week by diluting 0.5 mL of the LCS working spike stock to a final volume of 50 mL as summarized in the table below.

The LCS for batches of aqueous samples is prepared by adding 1.0 mL of the LCS spike solution to one liter of reagent water. The LCS for batches of soil samples is prepared by adding 1.0 mL of the LCS spiking solution to 30 g of Ottawa sand.

LCS Spiking Solution

Volume of AB Mix Stock (mL)	Conc of AB Mix Stock (µg/mL)	Final Volume (mL)	Final Concentration (µg/mL)
0.5	50	50	0.5

7.12 Matrix Spike (MS) Spike Solution, 0.5 µg/mL

The working matrix spike solution is the same as the LCS spike solution (Section 7.11). Matrix spikes (MS and MSD) are prepared by adding 1.0 mL of the working spike solution to one liter of an aqueous sample or to a 30-gram soil subsample.

7.13 Toxaphene Spike Solution, 2.0 µg/mL

7.13.1 A Toxaphene stock standard solution at a concentration of 1,000 µg/mL is purchased from commercial sources. This must be from a different source than is used for the initial calibration.

7.13.2 The working Toxaphene spike solution is prepared in a 500 mL volumetric flask by adding 1.0 mL of the stock solution (Section 7.13.1) and diluting to volume with acetone.

7.13.3 Aqueous LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 1.0 liter of reagent water. Soil LCSs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 30 grams of Ottawa sand.

7.13.4 Aqueous MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 1.0 liter of the selected aqueous sample. Soil sample MS/MSDs are prepared by adding 1.0 mL of the Toxaphene spike solution (Section 7.13.2) to 30 grams of the selected soil subsample.

7.14 Surrogate Spike Solution, 0.2 µg/mL

7.14.1 The surrogate stock solution, containing 200 µg/mL each of decachlorobiphenyl and tetrachloro-*m*-xylene (TCMX), is purchased from commercial sources.

7.14.2 The working surrogate spike solution is prepared in a 500 mL volumetric flask by adding 0.5 mL of the stock solution (Section 7.14.1) and diluting to volume with acetone.

7.14.3 For aqueous sample batches, 1.0 mL of the surrogate spike solution (Section 7.14.2) is added to each one-liter sample and QC sample. For soil sample batches, 1.0 mL of the surrogate spike solution (Section 7.14.2) is added to each 30-gram soil subsample and QC sample matrix.

7.15 Column Degradation Mix (EVAL B) (8081EvalBStk2)

7.15.1 The DDT/Endrin breakdown stock standard solution is obtained from commercial sources, with endrin at a concentration of 200 µg/mL, and 4,4'-DDT at 200 µg/mL (Accustandard M-8081-DS).

7.15.2 The working EVAL B solution is prepared in a 100 mL volumetric flask, by diluting 0.2 mL of the stock solution (Section 7.15.1) in hexane, as summarized in the following table:

Column Degradation Mix (Eval B Std) Spike Solution

Compound	Volume of Stock (mL)	Final Volume (mL)	Final Concentration (µg/mL)
Endrin	0.2	100	0.04
4,4'-DDT			0.04

7.16 Internal Standard Spiking Solution (8081_IS)

The spiking stock (BNB stock) at 2 µg/mL is prepared by diluting 0.2 mL of the commercial Internal Standard Stock from section 7.4.6 to a final volume of 100 mL in hexane. Every standard, QC sample, and client sample is spiked with 15 µL of the internal standard spiking solution into 0.20 mL. This produces a concentration of 0.150 ng/mL of internal standard in each sample. For the LVI method use half of the volume of internal standard spike (7.5 µL).

7.17 Primer Mix

The concentration of the column primer mix is not critical. It generally consists of a mixture of CCV, old ICAL standards, and/or old soil LCS extracts. The primer mix is used to initialize the column and does not affect calibration or quantitation.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples are collected in pre-cleaned, amber glass bottles fitted with a Teflon-lined cap. To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes. For the LVI option 250 mL of sample is needed.

8.2 Soil samples are collected in 8-ounce, pre-cleaned, wide-mouth jars with a Teflon-lined lid.

8.3 Samples are stored at ≤ 6 °C and not frozen.

8.4 Extracts are refrigerated at ≤ 6 °C.

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Waters	Amber glass	1 Liter 250 mL (for LVI)	Cool, <6°C, not frozen	7 Days	40 Days from extraction	SW-846
Soils	Glass	30 grams	Cool, <6°C, not frozen	14 Days	40 Days from extraction	SW-846

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal

Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on an instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13.0 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The method blank must be run on each instrument that is used to analyze samples from the same preparation batch. See QC Policy DV-QA-003P for further details.

9.4 Method Blank (MB)

At least one method blank must be processed with each preparation batch. The method blank for batches of aqueous samples consists of 1.0 liter of reagent water (the LVI method will require a 250 mL volume of reagent water), and for batches of soil samples, consists of 30 grams of Ottawa sand, both of which are free of any of the analyte(s) of interest. The method blank is processed and analyzed just as if it were a field sample.

Acceptance Criteria: The result for the method blank must be less than one-half the reporting limit for the analyte(s) of interest. For DoD QSM 4.2, QSM 5.0 or SW846 Method 8000D Revision 5 the acceptance criteria is no analytes detected > ½ RL

(i.e. LOQ) or > 1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater.

Corrective Action: If target analytes in the blank exceed the acceptance limits, the source of the contamination must be investigated. All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If the analyte was not detected in the samples, then the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

See Policy DV-QA-003P and Policy DV-QA-024P for further details.

9.5 Laboratory Control Sample (LCS)

At least one LCS must be processed with each preparation batch. For aqueous sample batches, the LCS consists of reagent water to which the analyte(s) of interest are added at a known concentration. For soil sample batches, the LCS consists of reagent sand to which the analyte(s) of interest are added at a known concentration. See Section 7.11 for the preparation of LCSs. The LCS is carried through the entire analytical procedure just as if it were a sample.

Acceptance Criteria: The recovery results for the LCS must fall within the established control limits. Control limits are set at ± 3 standard deviations around the historical mean. Where required, project-specific limits may be used in place of historical limits. Current control limits are maintained in the LIMS.

Note: For SC DHEC compliance samples, LCS recoveries must fall within 70-130%

When there are more than 11 analytes in the LCS, then NELAC allows a specified number of results to fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed MEs
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1

# of Analytes in LCS	# of Allowed MEs
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QAS's in the public folders for the current requirements.

Corrective Action: If LCS recoveries are outside of the established control limits, and the MS/MSD recoveries are also out of control limits then the system is out of control and corrective action must occur. If recoveries are above the upper control limit and the analyte(s) of interest is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed. If instrument maintenance and recalibration is performed and the LCS is reanalyzed as a corrective action for out of control LCS then all of the associated samples in the batch must also be reanalyzed.

9.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

One MS/MSD pair should be processed with each preparation batch. If sufficient sample is not available for an MS/MSD then a duplicate LCS should be prepared to establish precision. For DoD QSM 4.2 or QSM 5.0, the MS/MSD must be from the project site and if insufficient sample is available to analyze the MS/MSD pair, this is documented in an NCM but no LCSD is performed. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses a real sample matrix in place of the blank matrix. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked exactly as the MS) that is prepared and analyzed along with the sample and matrix spike. Refer to Section 7.12 for preparation of matrix spikes. Some programs allow spikes to be reported for project-related samples only. Samples identified as field blanks cannot be used for the MS/MSD analysis.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at $\pm$ 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD

control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD QSM 5 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being

flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.7 Surrogate Spikes

Every calibration standard, field sample, and QC sample (i.e., method blank, LCS, LCSD, MS, and MSD) is spiked with DCB and TCMX surrogate compounds. Refer to Section 7.14 for preparation of the surrogate spike solution.

Acceptance Criteria: The recovery of each surrogate must fall within established statistical limits, which are set at ± 3 standard deviations around the historical mean.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are acceptable. Low surrogate recoveries in the blank require re-preparation and reanalysis of the associated samples, unless sample surrogate recoveries are acceptable and targeted compounds are not detected.

For field samples, surrogate recoveries are usually calculated and reported for DCB only. TCMX may also be added. If two surrogate compounds are analyzed and

recoveries calculated, and either surrogate fails to meet acceptance criteria, corrective actions are required. (This also applies to programs that require the use of only one surrogate.) At least one surrogate must pass on any column from which target analytes are identified and reported or corrective action must be identified.

If surrogate recoveries fail, verify calculations, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference, which can be confirmed by examining the sample chromatogram, or due to the sample concentrating due to evaporation or improper adjustment of the final extract volume. Low recoveries may be due to adsorption by the sample matrix (i.e., clay particles, peat or organic material in the sample). Recalculate the data and/or reanalyze the extract if the checks reveal a problem.

If matrix interference is not obvious from the initial analysis, it is necessary to re-prepare / reanalyze a sample only once to demonstrate that poor surrogate recovery is due to a matrix effect, as long as it can be shown that the analytical system was in control. All out of control surrogates and associated corrective actions must be documented in an NCM.

9.8 Internal Standard

Acceptance Criteria: The internal standard recoveries for the opening CCVs for each 12 hour sequence must be within -50% to +100% of the response established by the midpoint of the ICAL. The internal standard response for the samples is compared to the most recent (preceding) calibration standard and must be within -50% and +100% of the response measured for that standard.

The internal standard in the must elute within 30 sec of the retention time window measured in the midlevel initial calibration point in the CCV and within 30 sec of the retention time of the initial CCV in the sequence in samples.

Corrective Action: If the internal standard response is outside of this range then the samples must be diluted until the recoveries are in control. Failure to meet this criteria in a CCV requires reanalysis of the standard and all affected samples analyzed in the bracket previous to the standard and after the standard. Recalibration is necessary if control cannot be established.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003 *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training.*

10.1.2 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Table 2 for typical operating conditions.

10.1.3 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the ChemStation software to set up the analytical sequence.

10.1.4 Unprocessed calibration data are transferred to the Chrom database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist, GC and HPLC ICAL TALS Review Checklist. (See SOP DV-QA-0020.) Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration review checklist is stored in the documents section of each analytical batch in TALS.

10.2 Column Degradation Evaluation

10.2.1 Each day of operation before any calibration or calibration verification standards are analyzed and at the beginning of each 12-hour shift, the column degradation evaluation mix (EVAL B) must be analyzed. The degradation check must be performed whether or not DDT, endrin, or degradation compounds are designated as target analytes. The purpose of the evaluation is to determine whether instrument/column maintenance is needed. The preparation of this standard is described in Section 7.15.

10.2.2 The results of the analysis of the EVAL B standard solution are used to calculate column degradation in terms of DDT percent breakdown (%B) and Endrin %B as follows:

$$\text{DDT } \%B = \frac{A_{DDD} + A_{DDE}}{A_{DDD} + A_{DDE} + A_{DDT}} \times 100\% \qquad \textit{Equation 1}$$

Where A_{DDD}, A_{DDE}, and A_{DDT} are the peak responses for 4,4'-DDD, 4,4'-DDE, and 4,4'-DDT, respectively, in the EVAL B chromatogram.

$$\text{Endrin } \%B = \frac{A_{EK} + A_{EA}}{A_{EK} + A_{EA} + A_{E}} \times 100\% \qquad \textit{Equation 2}$$

Where A_{EK}, A_{EA}, and A_E are the peak responses for endrin ketone, endrin aldehyde, and endrin, respectively, in the EVAL B chromatogram.

10.2.3 Acceptance Criteria

The %B for each of these two compounds, DDT and endrin, must not be greater than 15%.

10.2.4 Corrective Action

If the breakdown of DDT and/or endrin exceeds the 15% limit, corrective action must be taken. This action may include any or all of the following:

- Replacing the injection port liner or the glass wool.
- Cutting off a portion of the injection end of the column or guard column.
- Replacing the GC column or guard column
- Replacing the y-splitter.

After taking the appropriate corrective action, the degradation evaluation standard must be reanalyzed and must pass acceptance criteria before conducting any calibration events.

10.3 The laboratory uses six calibration levels (as shown in Table 3) for the single-component pesticides. The lowest point on the calibration curve is at or below the reporting limit (RL). The highest standard defines the highest sample extract concentration that may be reported without dilution. The preparation of the calibration standards is described in Section 7.6.

10.4 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.5 Calibration for the multi-peak component analytes, Toxaphene and Technical Chlordane, begins with a single-point calibration at or near the RL. If any multi-peak components are found to be present in the samples, a calibration for the multi-component analyte(s) is conducted with a minimum of five calibration levels. The samples are then reanalyzed using the full calibration curve that brackets the quantitation range.

10.6 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

10.7 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.7.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.7.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.7.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.7.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.8 If a data point is rejected, it must be documented in the sequence log and on an NCM which is filed with the project.

NOTE: Second order curves are not allowed for South Carolina work.

10.9 Internal Standard Calibration

Internal standard calibration involves the comparison of an instrument response (e.g., peak area or peak height) from the target compound in the sample to the response of the internal standard compound, which is added to the sample or sample extract prior to injection. See section 7.4 for the internal standards used. The same concentration of internal standard is added to each initial calibration standard. For each calibration level, the response factor, RF, is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$ *Equation 1*

Where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

10.10 Establishing the Calibration Function

Calibrations are modeled either as average response factors or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met.

10.10.1 Linear Calibration Using Average Response Factor

The response factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

10.10.1.1 The average response factor is calculated as follows:

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

Equation 2

Where:

RF_i = The response factor for the ith calibration level.

n = The number of calibration levels.

10.10.1.2 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\%$$

Equation 3

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(RF_i - \overline{RF}\right)^2}{n-1}}$$

Equation 4

10.10.2 Evaluation of the Average Response Factor

Plot the calibration curve using the average RF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., DoD, Arizona and South Carolina require evaluation of each compound individually). Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.10.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \text{Equation 5}$$

Where:

y = Instrument response (peak area or height).
x = Concentration of the target analyte in the calibration standard.
a = Slope of the line.
b = The y-intercept of the line.

For an internal standard calibration, the above equation takes the following form:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b \qquad \textit{Equation 6}$$

To calculate the concentration in an unknown sample extract, the regression equations 5 and 6 are solved for concentration, resulting in the following equations, where x and C_s are now the concentration of the target analyte in the unknown sample extract:

$$x = \frac{y - b}{a} \qquad \textit{Equation 7}$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a} \qquad \textit{Equation 8}$$

10.10.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less

than the reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.99. DoD QSM 5.0 requires r^2 >0.99.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

10.10.5 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 9}$$

Where a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

Non-Linear calibrations are NOT allowed for South Carolina compliance work

10.10.6 Evaluation of Second-Order Regression Calibration

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria: The coefficient of determination (COD) must be (r^2) ≥ 0.990.

Second-order regressions should be the last option, and note that some programs (e.g., South Carolina) do not allow the use of second-order regressions. A second order model must not be used to avoid maintenance. Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is approximately < one-half of the lowest concentration standard reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem should be investigated and the instrument recalibrated.

10.10.7 Polynomial regression fits of third order or higher are not allowed for this method.

10.10.8 Acceptance criteria independent of calibration model

10.10.8.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$\%\ Error = \frac{xi - xi}{xi} x\ 100$$

Where: $x'i$ = measured amount of the analyte at calibration level *i* in mass or concentration units and xi = the true amount of the analyte at calibration level *i* in mass or concentration units.
% Error should be ≤ 30%. For some data uses ≤ 50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error:

$$RSE = 100\,x\sqrt{\sum_{i=1}^{n}\left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

Where: *Xi* = True amount of the calibration level I, in mass or concentration units
X'i = measured amount of analyte in calibration level I, in mass or concentration units
p = number of terms in the fitting equation (average = 1), linear – 2, quaratic – 3)

n = number of calibration points
RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. IF not defined in the method, use ± 20%.

10.11 Initial Calibration Verification (ICV), 0.025 µg/mL for most compounds

A mid-level standard that is obtained from a source different from that of the calibration standards (second-source standard) is used to verify the initial calibration (see Section 7.8). The ICV standard is analyzed immediately following the initial calibration (ICAL).

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% for Method 8081A and ± 20% of the expected value(s) for Method 8081B.

Corrective Action: If the applicable criteria is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.12 Calibration Verification

10.12.1 12-Hour Calibration Verification

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.12.1.1 Continuing Calibration Verification (CCV), 0.05 µg/mL for most compounds.

NOTE: Arizona and Wisconsin require that the CCV concentration be varied throughout the sequence when calibration fits other than average response are used.

It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7.9).

At a minimum, this is analyzed after every 12 hours. Some programs (e.g., DOD) require analysis of a bracketing CCV which must be analyzed within 12 hours of the DDT/Endrin breakdown standard.

If 12 hours elapse, analyze the 12-hour standard sequence instead (including the Column Degradation Evaluation). Depending upon the program a closing CCV is not required when using an internal standard. DoD and Arizona require a bracketing CCVs.

NOTE: If a bracketing CCV is performed, the acceptance criteria in Section 10.12.3 apply to all samples.

10.12.2 RL Standard

It may also be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method at the end of the analytical sequence, as a minimum (see Section 7.10). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria.

10.12.3 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.12.3.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration must be within ± 15% for Method 8081A and ± 20% for Method 8081B using method 8000C criteria as a reference.

DoD QSM 5.0 requires recalibration and reanalysis of all affected samples since the last acceptable CCV. As an alternative, the laboratory may analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, the samples may be reported without reanalysis. If either fails, take corrective action(s) and recalibrate: then reanalyze all affected samples since the last acceptable CCV.

If a DoD client accepts TestAmerica's Technical Specifications for DoD QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags required by the project.

In some cases, the nature of the samples being analyzed may be the cause of the failing %D. When the %D for an analyte falls outside of the CCV criteria stated above, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed (at a

dilution if column damage is imminent) to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client.

Refer to Section 12 for which result to report.

In cases where additional compounds are to be analyzed in conjunction with compounds defined by this method and that are not defined in the scope and application of method 8081B different CCV acceptance criteria may apply. Kepone is not recommended by method 8081B and the CCV acceptance criteria is defined as +/- 53%. Further these additional compounds will not be used in grand mean calculations (when applicable) as discussed below.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \textit{Equation 11}$$

10.12.3.2 Analytes Not Detected (< RL)

For any analyte not detected in client samples, the %D for that analyte in the bracketing CCVs should also be within ± 20% for Method 8081B or within 15% for Method 8081A. For method 8081B Test America Denver references method 8000C and 8000D for compounds with curve fits other than an average curve fit. See also DV-QA-027P for further evaluation criteria. Any deviation for the calibration criteria outlined in this procedure must be documented in an NCM.

NOTE: The grand mean must not be applied when Method 8000C or 8000D is applicable (e.g., Arizona)

10.13 Retention Time Windows

10.13.1 Initial determination of Retention time windows.

10.13.1.1 The center of the retention time (RT) window shall be updated based on the middle level standard in the initial calibration or the first CCV in the daily analytical sequence, whichever is more recent.

10.13.1.2 Evaluate the deviation from expected retention time for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours.

10.13.1.2.1 If three days of analytical data are not available, use a default RT window of 0.01 minute. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

10.13.1.3 Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at 0.01 min. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

10.13.1.4 Retention time windows for analytes of interest must not overlap.

10.13.2 Ongoing evaluation of retention time windows

10.13.2.1 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the analytical sequence. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

10.13.2.2 Matrix spike analytes may fall outside the retention time window if there is a large non-target peak coeluting with the analyte in the matrix spike.

10.13.2.3 If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

10.13.2.4 Retention time windows should be reliably narrower than ± 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

10.13.3 Determine new RT windows each time a new column is installed, after a major analysis revision or maintenance event, or annually, whichever is most frequent.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be check for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 The final extract volume in hexane is 10 mL. The LVI method final volume is 5 mL.

11.3.5 Use hexane to dilute sample extracts, if necessary.

11.4 Instrument Maintenance and Troubleshooting

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance. Typically for the 8081 analysis the injection port liner must be changed daily in order to facilitate a passing DDT/Endrin breakdown standard. If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control. The injector septum should be changed after about 200 injections have been completed. If the last CCV that was analyzed indicated a high response then a simple liner change is typically sufficient to bring the system back into control. Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column. A reduced response may indicate that the system needs to be evaluated for leaks. Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed. Re-calibration should

not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.5 Gas Chromatography and Resolution

11.5.1 Chromatographic conditions for this method are presented in Table 2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the Chrom database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the Chrom software.

11.5.2 The Pesticides Minimum Requirements states that there should be at least 80% resolution for a given analyte on at least one column. The chromatographic conditions must be set in a way to maintain this requirement.

11.6 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. Samples, standards, and QC samples must be introduced using the same procedure. Use the ChemStation interface to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.7 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Table 3 for the calibration levels used.

11.7.1 Prior to analyzing any calibration or calibration verification standards, the column degradation evaluation standard is injected and the results are evaluated as described in Section 10.2.

11.7.2 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.12.1) and updating the retention time windows (see Section 10.13).

11.7.3 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification.

11.7.4 The following is a typical analytical sequence:

- Primer
- Hexane blank
- Eval B Std (column degradation evaluation)
- Daily initial CCVs
- LCS

- Method Blank
- Samples for up to 12 hours after the Eval B (or 20 injections of samples and QC samples, whichever is more frequent)
- CCVs (within 12 hours of Eval B Std for closing requirement*)
- Eval B Std (column degradation evaluation)
- Followed by 12 hour cycles of samples and CCVs as needed
- Closing CCV (if required by program).

*DoD requires that CCVs are analyzed every 10 field samples.

11.8 Daily Retention Time Windows

The centers of the retention time (RT) windows determined in Section 10.13 are adjusted to the RT of each analyte as determined in the 12-hour calibration verification. The centers of the RT windows must be updated at the beginning of each analytical sequence.

11.9 Manual Integration and Data Review

Upon completion of the analytical sequence, transfer the raw chromatography data to the CHROM database for further processing.

11.9.1 Review chromatograms online and determine whether manual data manipulations are necessary.

11.9.2 All manual integrations must be justified and documented. See DV-QA-011P requirements for manual integration.

11.9.3 Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature.

11.9.4 Alternatively, the manual integration may be processed manually. In the latter case, print both the before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. The manually processed chromatograms must be scanned and attached to the project in TALS.

11.10 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.10.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.10.2 Perform a level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.10.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the level 2 review checklist. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Qualitative Identification

12.1.1 Tentative identification of an analyte occurs when a peak is found on the primary column within the RT window for that analyte, at a concentration above the reporting limit, or above the MDL if qualified data (J flags) are to be reported. Identification is confirmed if a peak is also present in the RT window for that analyte on the second (confirmatory) column and if the analyte concentration is greater than the MDL. When confirmation is made using a second column, the analysis on the second column must meet all of the QC criteria for continuing calibration verification and RTs.

12.1.2 The experience of the analyst should weigh heavily in the interpretation of the chromatogram. For example, sample matrix or laboratory temperature fluctuation may result in variation of retention times. If a RT shift greater than the RT window occurs for a reported compound the situation must be explained in an NCM.

12.2 Dual-Column Quantitation and Reporting

12.2.1 A primary column is designated. The result from the primary column is normally reported. If the continuing calibration verification fails on one of the columns, the appropriate corrective action must be taken. The result from the secondary (confirmation) column may be reported if either of the following possibilities are true:

12.2.1.1 There is obvious chromatographic interference on the primary column.

12.2.1.2 The result on the primary column is > 40% greater than the result on the secondary column.

12.2.2 For DoD QSM 4.2 or QSM 5.0 work, calibration and QC criteria for the second column are the same as for the initial or primary column analysis.

12.2.3 Dual Column Results With >40% RPD

12.2.3.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram.

12.2.3.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.2.3.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.2.3.4 The RPD between two results is calculated using the following equation:

$$RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the primary column and R_2 is the result for the confirmation column.

12.3 Multi-Component Analytes (Toxaphene and Technical Chlordane)

12.3.1 Qualitative Identification

Retention time windows are also used for identification of multi-component analytes, but the "fingerprint" produced by major peaks of those compounds in the standard is used in tandem with the retention times to identify the compounds. The ratios of the areas of the major peaks are also taken into consideration. Identification of these compounds may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram.

12.3.2 Quantitation of Toxaphene

12.3.2.1 While Toxaphene contains a large number of compounds that produce well resolved peaks in a GC/ECD chromatogram, it also contains many other components that are not chromatographically resolved. The unresolved complex mixture results in a "hump" in the chromatogram that is characteristic of the Toxaphene mixture of compounds. The resolved peaks are important for the identification of the mixture, and the area of the unresolved complex mixture contributes a significant portion of the area of the total response.

12.3.2.2 To measure total area, construct the baseline of Toxaphene in the sample chromatogram between the RTs of the first and last eluting Toxaphene components in the standard. In order to use the total area approach, the pattern in the sample

chromatogram must be compared to that of the standard to ensure that all of the major components in the standard are present in the sample. Otherwise, the sample concentration may be significantly underestimated.

12.3.2.3 Toxaphene may also be quantitated on the basis of 4 to 6 major peaks. Using a subset of 4 to 6 peaks for quantitation provides results that agree well with the total peak approach and may avoid difficulties when interferences with Toxaphene peaks are present in the early portion of the chromatogram from compounds such as DDT. Construct the baseline as outlined in 12.3.2.2.

12.3.2.4 When Toxaphene is determined using the 4 to 6 peaks approach, care must be taken to evaluate the relative areas of the peaks chosen in the sample and standard chromatograms. The laboratory typically uses 5 peaks.

12.3.2.5 The chosen peaks must be within the established retention time. If there is an interference that affects the accuracy of results, the analyst may use as few as 4 major peaks. The same peaks that are used for sample quantitation must be used for calibration.

12.3.2.6 The heights or areas of the chosen peaks should be summed together and averaged to determine the Toxaphene concentration. Since toxaphene is a production mixture and does not necessarily match all possible mixtures, each of the individual peaks used to quantitate toxaphene are not controlled up. The average of the chosen peaks is what is controlled upon

12.3.2.7 Second column confirmation of multi-component analytes will be performed.

12.3.3 Quantitation of Technical Chlordane

12.3.3.1 Technical Chlordane is a mixture of at least 11 major components and 30 or more minor components that is used to prepare specific pesticide formulations. *cis*-Chlordane (or α-Chlordane) and *trans*-Chlordane (or γ-Chlordane) are the two most prevalent major components of Technical Chlordane. However, the exact percentage of each in the technical material is not completely defined, and is not consistent from batch to batch.

12.3.3.2 When the GC pattern of the sample resembles that of Technical Chlordane, Chlordane may be quantitated by comparing the total area of the Chlordane chromatogram or by using 3 to 5 major peaks. Typically the laboratory uses 5 peaks. Since technical chlordane is a production mixture and

does not necessarily match all possible mixtures, each of the individual peaks used to quantitate technical chlordane are not controlled up. The average of the chosen peaks is what is controlled upon.

12.3.3.3 If the Heptachlor epoxide peak is relatively small, include it as part of the total Chlordane area for calculation. If Heptachlor and/or Heptachlor epoxide are much out of proportion, calculate these separately and subtract their areas from the total area to give a corrected Chlordane area.

12.3.3.4 To measure the total area of the Chlordane chromatogram, construct the baseline of Technical Chlordane in each calibration chromatogram between the RTs of the first and last eluting Technical Chlordane components. Use this area and the mass or concentration of Technical Chlordane in each calibration standard to establish the calibration function (Section 10.0). Construct a similar baseline in the sample chromatogram, measure the area, and use the calibration function to calculate the concentration in the sample extract.

12.3.3.5 When the GC pattern of Chlordane in a sample differs considerably from that of the Technical Chlordane standard, it may be practical to report "Chlordane (not otherwise specified, CAS number 57-74-9)." Using the same process and calibration as for reporting Technical Chlordane.

12.3.3.6 A third option for quantitating Technical Chlordane is to quantitate the peaks for α-Chlordane, γ-Chlordane, and Heptachlor separately against the appropriate reference materials, and report these individual components under their respective CAS numbers.

12.3.3.7 Second column confirmation of multi-component analytes will be performed.

12.4 Surrogate recovery results are calculated and reported for DCB. TCMX may also be added, however if the two surrogate compounds are analyzed, and recoveries are calculated, and either surrogate fails to meet control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). See section 9.7 for further details.

12.5 Calibration Range and Sample Dilutions

12.5.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted with hexane (record the hexane lot number in the run sequence) and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the

RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e. the sample's concentration may be similar to or higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.5.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.5.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.5.4 Reporting Dilutions

Some programs (e.g., South Carolina) and some projects require reporting of multiple dilutions (check special requirements in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported. When reporting multiple dilutions, unless otherwise requested, the analyst typically reports the lowest dilution practical (one that is not obscured by the matrix) and then one or two higher dilutions so that the bulk of the detections are quantifiable and all of the compounds are within the calibration range.

12.6 Interferences Observed in Samples

12.6.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.6.2 Suspected Negative Interferences

12.6.2.1 If peak detection is prevented by interferences, further cleanup should be attempted (see SOP DV-OP-0007). Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.6.2.2 If the individual isomers of chlordane are identified, but there is no pattern for the confirmation of "Technical Chlordane", and the project has ONLY technical chlordane requested, the results for technical chlordane should be qualified to indicate the presence of the chlordane isomers. Document in a NCM.

12.6.3 Suspected Positive Interferences

12.6.3.1 If the technical chlordane pattern is present, but only the individual isomers are requested to be reported, document in a NCM.

12.6.3.2 If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option, however caution is warranted in order to rule out false negatives. At a minimum, an NCM should be prepared by the analyst and should include the following comment for inclusion in the case narrative:

"*Based on review of the chromatograms for samples __, it is my opinion that the evident interferences may be causing false results.*

Date _____ Analyst _____"

Sample dilution may be the only acceptable recourse to resolve detections when large amounts of non-target matrix are observed.

12.7 Calculations

12.7.1 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 15}$$

12.7.2 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD}\ \%\text{Recovery} = \left(\frac{SSR - SR}{SA}\right) \times 100\% \qquad \textit{Equation 16}$$

Where:

SSR = Measured concentration in spiked sample.
SR = Measured concentration in unspiked sample.
SA = Concentration of spike added to sample.

12.7.3 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 17}$$

Where R_1 is the result for the MS and R_2 is the result for the MSD.

12.7.4 Concentration of Analyte in the Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.0 for details on establishing the calibration function):

Average Response Factor: $C_e = \frac{A_s}{RF}$ *Equation 18*

Linear Regression: $C_e = \frac{[A_s - b]}{a}$ *Equation 19*

Non-Linear Regression: $C_e = f(A_s)$ *Equation 20*

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).
A_s = Peak area for the analyte in the sample extract injection.
b = y-intercept of the calibration fit.
a = Slope of the calibration fit.
$f(A_s)$ = Mathematical function established by the non-linear regression.

12.7.5 Concentration of Analyte in Original Sample (for 1 uL injection)

$$C_{sample} = \frac{C_e}{1000\,\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \textit{Equation 21}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e	=	Concentration of analyte in sample extract injected in GC (ng/mL).
$1000\ \frac{ng}{\mu g}$	=	Factor to convert ng/mL to µg/mL.
V_e	=	Volume of sample extract (mL).
V_s	=	Volume (or weight) of original sample (L or kg).
DF	=	Dilution Factor (post extraction dilutions)

12.8 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13.0 Method Performance

13.1 Method Detection Limit (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 MDLV and LOQV

13.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes the programmatically required spike amounts.

13.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

13.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

13.3 Demonstration of Capabilities

IDOCs must be performed initially, before samples are analyzed, and must be repeated on an annual basis. Each analyst performing this procedure must successfully analyze four LCS QC samples using current laboratory LCS control limits before they are allowed to analyze samples without direct oversight. Prior to the successful completion and approval (by the QA department) of the IDOC, the supervising analyst must document this oversight by including a comment in the "notes" section of the worksheet in TALS.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Expired extract vial waste - Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the

Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.2 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.3 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.4 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.5 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.6 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

16.7 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.8 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.9 Method 8081A, Organochlorine Pesticides by Gas Chromatography, Revision 1, December 1996

16.10 Method 8081B, Organochlorine Pesticides by Gas Chromatography, Revision 2, February, 2007.

16.11 Method 8000B, Determinative Chromatographic Separations, Revision 2, December, 1996.

16.12 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.13 Method 8000D, Determinative Chromatographic Separations, Revision 5, March 2018

17.0 Method Modifications:

Item	Method	Modification
1	8081A 8081B	Section 10.5 allows the use of a single-point calibration for the multi-component pesticides. In this SOP an initial single-point calibration is used, but a five-point calibration followed by reanalysis of associated samples is required when one of the multi-component pesticides is detected.
2	8081A 8081B	Method 8081 references 8000, which allows the use of third-order calibration curves. TestAmerica Denver does not allow third-order curves.
3	8081A	Section 10.7.2 excludes the use of the grand average of % RSD

Item	Method	Modification
	8081B 8000B	and requires each compound meet % RSD criteria for the initial calibration while Method 8000B allows acceptance using the mean of % RSD for all compounds in the calibration.
4	8081A 8081B 8000B 8000C 8000D	Minimum retention time window (± 0.01 minute) is more stringent than the Method 8000B or 8000D window of ± 0.03 minute. The established window may be adjusted based on RT drift observed in the ICAL.
5	8081B	Section 11.7.2 – Use 8000D criteria for calibration verification when a non-average curve fit is used.

18.0 Tables and Attachments

Table 1: Analyte List and Standard Reporting Limits
Table 2: Typical Instrument Conditions
Table 3: Calibration Levels (µg/mL)
Table 4: LVI Method Calibration Levels (µg/mL)
Table 5: Column Degradation Evaluation Mix
Table 6: LCS/Matrix Spike and Surrogate Spike Levels
Table 7: Evaluation Criteria and Corrective Actions for Continuing Calibration Verification
Table 8: Chrom Peak Number Assignment for Analytes – Columns CLPI & CLPII
Table 9: Chrom Peak Number Assignment for Analytes – Columns RxiSil 35-MS & Rxi-XLB
Attachment 1: Example Chromatogram – Columns CLPI & CLPII – AB Standard
Attachment 2: Example Chromatogram – Columns CLPI & CLPII – AP9 Standard
Attachment 3: Example Chromatogram – Columns CLPI & CLPII – Chlordane (Technical)
Attachment 4: Example Chromatogram – Columns CLPI & CLPII – Toxaphene
Attachment 5: Example Chromatogram – Columns RxiSil 35-MS & Rxi-XLB – AB Standard
Attachment 6: Example Chromatogram – Columns RxiSil 35-MS & Rxi-XLB – AP9 Standard
Attachment 7: Example Chromatogram – Columns RxiSil 35-MS & Rxi-XLB – Chlordane (Technical)
Attachment 8: Example Chromatogram – Columns RxiSil 35-MS & Rxi-XLB – Toxaphene

19.0 Revision History

- Revision 16, dated 19 September 2019
  - Added a note to section 9.5 regarding LCS limit requirements for SC DHEC compliance samples.
- Revision 15, dated 04 January 2019
  - Added sections 13.2 and 3.5 in reference to LLOQ checks and standards.
- Revision 14, dated 31 October 2018
  - Added references to SW 846 Method 8000D
  - Added explanations and calculations for % Error and Relative Standard Error
  - Added references to South Carolina compliance.
  - Corrected TestAmerica's name and copyright information.
  - Updated MDL SOP number

- Revision 13, dated 31 August 2018
  - Annual Review
- Revision 12, dated 31 October 2017
  - Added statement that we control upon the average of the chosen peaks to section 12.3.2.6 and 12.3.3.2.
- Revision 11, dated 31 August 2016
  - Added propachlor to standards and compound lists throughout
  - Corrected LVI volume for Method 3510C
  - Revised MS/MSD corrective actions in Section 9.6 to meet current policy requirements
  - Changed use of term from Calibration Factor to Calibration Response to account for change to internal standard method of calibration for instances missed in last revision
  - Added Sections 10.10.5 and 10.10.6
  - Revised frequency of CCV in Section from 20 samples to 12 hours to reflect current practice.
  - Revised Section 10.13 for determination of retention time windows to reflect current corporate policy
  - Revised analytical sequence in Section 11.7.4 to reflect current practice.
  - Removed statements in Section 12 for single column analysis as laboratory now always performs dual column confirmation on all analytes
  - Replaced references to "CLD" qualifier with use of NCM.
  - Added Table 9
  - Added Attachments 5-8
  - Removed references to AFCEE throughout
- Revision 10, dated 31 July 2015
  - Added use of Internal Standard (1-bromo-2-nitrobenzene) throughout. Calibration changed from external standard to internal standard (Sections 7, 10.9, 10.10). Changed "calibration factor" to "response factor" throughout.
  - Removed conflicting text for MB acceptance in DOD program in corrective action section of Section 9.4.
  - Updated SOP reference in Section 10.1.1 to revised corporate document number.
  - Revised section 10.12 to eliminate redundant language
  - Revised Section 10.13.7 to correct minimum RT window to $\pm$ 0.03 from $\pm$ 0.01. The latter is the minimum standard deviation to be used.
- Revision 9, dated 31 October 2014
  - Added instrument model numbers in Section 6.1
  - Identified where columns are used, by instrument in Section 6.4
  - Added more information regarding GC supplies in Section 6.6
  - Updated network location references to address current practice
  - Added TALS standard IDs throughout section 7
  - Added propachlor to the analyte list for water and throughout the SOP as needed.
  - Added criteria for DoD QSM 5.0 throughout
- Revision 8, dated 31 October 2013

- Formatting updates
- Section 1.3 – Removed reference to microwave LVI extractions
- Section 2.1.1. 9.4 – Listed appropriate information for LVI procedure
- Section 2.1.3, 4.1, 7.15.1, 7.15.2 (table), 10.1.14, 10.12.4.1, 10.12.4.2, 11.3.4, 11.5, 12.3.3.4, 12.5.4, Table 2, Table 3 (AP9 Standards), Table 4 (Chlordane Technical & Toxaphene, AP9 Standards, Surrogates), Table 5 – Added details to reflect current practices
- Section 7.4 – Revised standard mix information
- Section 7.5.1 – Updated table
- Section 7.6.2 – Added detail and calibration levels
- Section 7.6.4 – Added level 1 calibration standard and updated subsequent levels
- Section 7.7 & 7.8 – Revised Standard detail and update section tables
- Section 13 – Update MDL and IDOC/DOC information
- Section 17 – Added item number 6 in the method modification table
- Added table 8 – Chrom Peak and Peak Numbers
- Updated attachments with Chrom chromatograms for AB mix, AP9 mix, Chlordane (Technical) and Toxaphene as attachments 1 – 4.

- Revision 7.0, dated 12 October 2012
  - Added section 1.5 to state that the LVI procedure is not approved by South Carolina
- Revision 6.0, dated 16 July 2012
  - Corrected grammatical and formatting errors.
  - Added the information on the LVI procedure throughout the SOP
  - Added paragraph on "reagent grade" materials to Section 7
  - Added Section 11.4 – Instrument Maintenance
  - Updated Table 1 to include LVI information
  - Added Table 3
- Revision 5.0, dated 30 June 2011
  - Combines SOP No. DV-GC-0020 and SOP No. DV-GC-0026, superseding the latter, implemented 28 February 2011.
  - Updated equipment and supplies section
  - Aligned language with other GC SOPs for clarity and consistency in calibration and data review sections
  - Updated standards and reporting limits table.
  - Revised reporting criteria in Section 12.2

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List and Standard Reporting Limits

Compound	Water Reporting Limit (µg/L) [1 L sample]	Water Reporting Limit (µg/L) [LVI]	Soil Reporting Limit (µg/kg)
Aldrin	0.05	0.05	1.7
α-BHC	0.05	0.05	1.7
β-BHC	0.05	0.05	1.7
δ-BHC	0.05	0.05	1.7
γ-BHC (Lindane)	0.05	0.05	1.7
α-Chlordane	0.05	0.05	1.7
γ-Chlordane	0.05	0.05	1.7
Chlordane (technical)	0.5	0.5	25
Chlorobenzilate*	0.10	0.10	30
Chlorpyrifos*	0.05	0.1	–
DBPP***	2.50	2.50	140
2,4'-DDD*	0.05	0.05	0.33
4,4'-DDD	0.05	0.05	1.7
2,4'-DDE*	0.05	0.05	0.33
4,4'-DDE	0.05	0.05	1.7
2,4'-DDT*	0.05	0.05	0.33
4,4'-DDT	0.05	0.05	1.7
Diallate*	1.0	5.0	33
Dicofol*	1.0	10.0	–
Dieldrin	0.05	0.05	1.7
Endosulfan I	0.05	0.05	1.7
Endosulfan II	0.05	0.05	1.7
Endosulfan Sulfate	0.05	0.05	1.7
Endrin	0.05	0.05	1.7
Endrin Aldehyde	0.05	0.05	1.7
Endrin Ketone	0.05	0.05	1.7
Heptachlor	0.05	0.05	1.7
Heptachlor Epoxide	0.05	0.05	6.7
Hexachlorobenzene	0.05	0.05	1.7
Isodrin	0.10	0.10	1.7
Kepone**	1.0	1.0	75
Methoxychlor	0.10	0.10	3.3
Mirex	0.05	0.05	1.7
Propachlor	0.5	0.5	-
Toxaphene	2.0	2.0	67

* These are non-routine compounds that require a separate calibration, and are analyzed only upon request.

** The laboratory has some clients with permits requiring kepone by method 8081A and 8081B. However, the method warns that kepone may change form during extraction and shift out of the expected retention time window. Kepone is not recommended by 8081A and 8081B.

*** Available for analysis by method 8081A only.

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions*
Injection port temperature	200 °C
Detector temperature	325 °C
Column 1 (HP6890 GC)	Rtx® CLPI: 30 m X 0.32 mm id, 0.5 µm
Column 2 (HP6890 GC)	Rtx®CLPII: 30 m X 0.32 mm id, 0.25 µm
HP6890 GC Temperature program and inlet pressure Columns 1 and 2	110 °C for 1 minute 35 °C/min to 180 °C 20 °C/min to 200 °C 35 °C/min to 235 °C and hold for 1 minute 25 °C/min to 300 °C and hold for 4 minutes 40 °C/min to 310 °C Pressure 20 psi, pulse to 40 psi for 1 minute
Column 3 (HP6890 GC)	DB-35MS: 30 m X 0.32 mm id, 0.5 µm
Column 4 (HP6890 GC)	DB-XLB: 30 m X 0.32 mm id, 0.5 µm
HP6890 GC Temperature program Columns 3 and 4	110 °C for 1 minute 35 °C/min to 245 °C and hold for 1.5 minutes 25 °C/min to 300 °C and hold for 4 minutes 40 °C/min to 310 °C
Injection	1 or 2 µL (for LVI)
Carrier gas	Hydrogen
Make up gas	Nitrogen, 60 mL/min
Y splitter	Restek or J&W or Supelco glass tee (Siltek)

* Variations in instrument conditions may exist in order to facilitate compound separation or to accommodate matrix effects from sample analysis.

NOTE: 4,4'-DDE and dieldrin are closely eluting pairs on the HP-5 column . Endosulfan II and 4,4'-DDD are closely eluting pairs on the 1701 column. For these reasons, these columns are no longer in use in the laboratory.

Table 3. Calibration Levels (µg/mL)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Individual Mix AB						
Aldrin	0.004	0.01	0.025	0.05	0.075	0.10
α-BHC	0.004	0.01	0.025	0.05	0.075	0.10
β-BHC	0.004	0.01	0.025	0.05	0.075	0.10
δ-BHC	0.004	0.01	0.025	0.05	0.075	0.10
γ-BHC (Lindane)	0.004	0.01	0.025	0.05	0.075	0.10
α-Chlordane	0.004	0.01	0.025	0.05	0.075	0.10
γ-Chlordane	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDD	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDE	0.004	0.01	0.025	0.05	0.075	0.10
4,4'-DDT	0.004	0.01	0.025	0.05	0.075	0.10
Dieldrin	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan I	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan II	0.004	0.01	0.025	0.05	0.075	0.10
Isodrin	0.004	0.01	0.025	0.05	0.075	0.10
Endrin	0.004	0.01	0.025	0.05	0.075	0.10
Endrin Aldehyde	0.004	0.01	0.025	0.05	0.075	0.10
Endrin Ketone	0.004	0.01	0.025	0.05	0.075	0.10
Heptachlor	0.004	0.01	0.025	0.05	0.075	0.10
Heptachlor Epoxide	0.004	0.01	0.025	0.05	0.075	0.10
Hexachlorobenzene	0.004	0.01	0.025	0.05	0.075	0.10
Methoxychlor	0.004	0.01	0.025	0.05	0.075	0.10
Endosulfan Sulfate	0.004	0.01	0.025	0.05	0.075	0.10
Mirex	0.004	0.01	0.025	0.05	0.075	0.10
Multicomponent Standards						
Chlordane (Technical)	0.10	0.20	0.50	1.0	2.0	N/A
Toxaphene	0.20	0.50	1.0	2.0	5.0	10.0
Surrogates are included the AB Mix calibration mix at the following levels:						
Tetrachloro-*m*-xylene	0.005	0.10	0.025	0.05	0.075	0.10
Decachlorobiphenyl	0.005	0.10	0.025	0.05	0.075	0.10

Appendix IX Standards:							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2,4'-DDD	0.001	0.005	0.010	0.025	0.035	0.05	0.10
2,4'-DDE	0.001	0.005	0.010	0.025	0.035	0.05	0.10
2,4'-DDT	0.001	0.005	0.010	0.025	0.035	0.05	0.10
Chlorobenzilate	0.01	0.050	0.10	0.25	0.35	0.5	1.0
Chlorpyrifos	0.005	0.025	0.050	0.125	0.175	0.25	0.5
DBPP	0.50	0.250	0.5	1.25	1.75	2.5	5.0
Diallate	0.250	0.50	1.0	2.5	3.5	5	10.
Dicofol	0.01	0.050	0.10	0.25	0.35	0.5	1.0
Propachlor	0.01	0.050	0.10	0.25	0.35	0.5	1.0
Kepone	0.01	0.050	0.10	0.25	0.35	0.5	1.0

Table 4. LVI method. Calibration Levels (µg/mL)

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Individual Mix AB						
Aldrin	0.002	0.005	0.0125	0.025	0.0375	0.05
α-BHC	0.002	0.005	0.0125	0.025	0.0375	0.05
β-BHC	0.002	0.005	0.0125	0.025	0.0375	0.05
δ-BHC	0.002	0.005	0.0125	0.025	0.0375	0.05
γ-BHC (Lindane)	0.002	0.005	0.0125	0.025	0.0375	0.05
α-Chlordane	0.002	0.005	0.0125	0.025	0.0375	0.05
γ-Chlordane	0.002	0.005	0.0125	0.025	0.0375	0.05
4,4'-DDD	0.002	0.005	0.0125	0.025	0.0375	0.05
4,4'-DDE	0.002	0.005	0.0125	0.025	0.0375	0.05
4,4'-DDT	0.002	0.005	0.0125	0.025	0.0375	0.05
Dieldrin	0.002	0.005	0.0125	0.025	0.0375	0.05
Endosulfan I	0.002	0.005	0.0125	0.025	0.0375	0.05
Endosulfan II	0.002	0.005	0.0125	0.025	0.0375	0.05
Isodrin	0.002	0.005	0.0125	0.025	0.0375	0.05
Endrin	0.002	0.005	0.0125	0.025	0.0375	0.05
Endrin Aldehyde	0.002	0.005	0.0125	0.025	0.0375	0.05
Endrin Ketone	0.002	0.005	0.0125	0.025	0.0375	0.05
Heptachlor	0.002	0.005	0.0125	0.025	0.0375	0.05
Heptachlor Epoxide	0.002	0.005	0.0125	0.025	0.0375	0.05
Hexachlorobenzene	0.002	0.005	0.0125	0.025	0.0375	0.05
Methoxychlor	0.002	0.005	0.0125	0.025	0.0375	0.05
Endosulfan Sulfate	0.002	0.005	0.0125	0.025	0.0375	0.05
Mirex	0.002	0.005	0.0125	0.025	0.0375	0.05
Multicomponent Standards						
Chlordane (Technical)	0.025	0.1	0.25	0.50	1.0	2.0
Toxaphene	0.1	0.25	0.5	1.0	2.5	5.0
Appendix IX Standards:						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2,4'-DDD	0.0005	0.0025	0.005	0.0125	0.0175	0.025	0.05
2,4'-DDE	0.0005	0.0025	0.005	0.0125	0.0175	0.025	0.05
2,4'-DDT	0.0005	0.0025	0.005	0.0125	0.0175	0.025	0.05
Chlorobenzilate	0.005	0.025	0.05	0.125	0.175	0.25	0.5
Chlorpyrifos	0.0025	0.0125	0.025	0.0625	0.0875	0.125	0.25
DBPP	0.25	0.125	0.25	0.625	0.875	1.25	2.5
Diallate	0.125	0.25	0.5	1.25	1.75	2.5	5.0
Dicofol	0.005	0.025	0.05	0.125	0.175	0.25	0.5
Propachlor	0.005	0.025	0.05	0.125	0.175	0.25	0.5
Kepone	0.005	0.025	0.05	0.125	0.175	0.25	0.5
Surrogates are included the AB Mix calibration mix at the following levels:						
Tetrachloro-*m*-xylene	0.002	0.005	0.0125	0.025	0.0375	0.05
Decachlorobiphenyl	0.002	0.005	0.0125	0.025	0.0375	0.05

Table 5. Column Degradation Evaluation Mix

Component	Concentration (µg/mL)
4,4'-DDT	0.040
Endrin	0.040

Table 6. LCS/Matrix Spike and Surrogate Spike Levels

Compound	(µg/L)	(µg/kg)
Aldrin	0.5	16.67
α-BHC	0.5	16.67
β-BHC	0.5	16.67
δ-BHC	0.5	16.67
γ-BHC (Lindane)	0.5	16.67
α-Chlordane	0.5	16.67
γ-Chlordane	0.5	16.67
4,4'-DDD	0.5	16.67
4,4'-DDE	0.5	16.67
4,4'-DDT	0.5	16.67
Dieldrin	0.5	16.67
Endosulfan I	0.5	16.67
Endosulfan II	0.5	16.67
Endosulfan Sulfate	0.5	16.67
Endrin	0.5	16.67
Endrin Aldehyde	0.5	16.67
Endrin Ketone	0.5	16.67
Heptachlor	0.5	16.67
Heptachlor Epoxide	0.5	16.67
Methoxychlor	0.5	16.67
Toxaphene (when required)	2.0	66.68
Surrogates		
Decachlorobiphenyl	0.2	6.67
Tetrachloro-*m*-xylene (TCMX)	0.2	6.67

Table 7. Evaluation Criteria and Corrective Actions for Continuing Calibration Verification

Evaluation Criteria for a Specific Analyte				Evaluation / Corrective Actions
Average %D	Individual %D	RL Standard	Client Samples	
N/A	± 15%	N/A	≥ RL	Calibration is verified for the analyte(s) detected in the sample; no action required.
N/A	Outside of ± 15%	N/A	≥ RL	Calibration is not verified for the analyte(s) detected in the sample. The sample must be re-analyzed using a verified calibration.
± 15%	± 30%	N/A	ND	Calibration is acceptable because analytes were not detected in the sample. An NCM is required.
Outside of ± 15%	N/A	N/A	N/A	Calibration is not verified and corrective action must be taken. NOTE: The exception to this may be those cases where the client has requested a small subset of the analytes typically measured by the method and the %D for each of those analytes is within ± 15%. Corrective action may include clipping the column, changing the liner, or other minor instrument adjustments, followed by reanalyzing the standard twice. If both results pass acceptance criteria, the calibration may be used to process samples. If the overall average %D still varies by more than ±15%, a new calibration curve must be prepared. Reanalyze any samples that were either preceded by or followed by the failed CCV using a verified calibration.
± 15%	< -30% (low)	Detected	ND	Sample results are acceptable because the RL standard indicates that the analyte would have been detected if present in the sample. Explain in an NCM.
± 15%	< -30% (low)	ND	ND	Analyte was not detected in the RL standard, possibly as the result of a calibration drift in the negative direction, and therefore one cannot be sure that the analyte would have been detected in the sample if present. Reanalyze samples with verified calibration.
± 15%	> +30% (high)	N/A	ND	Sample results are acceptable because the CCV failed high, so if the analyte were present in the sample, it would definitely have been detected. Explain in an NCM.

Note: Some programs (e.g., South Carolina) do not allow the average percent difference to be used in evaluating calibration verification standards. Please see the QAS's in the public folders for the current requirements.

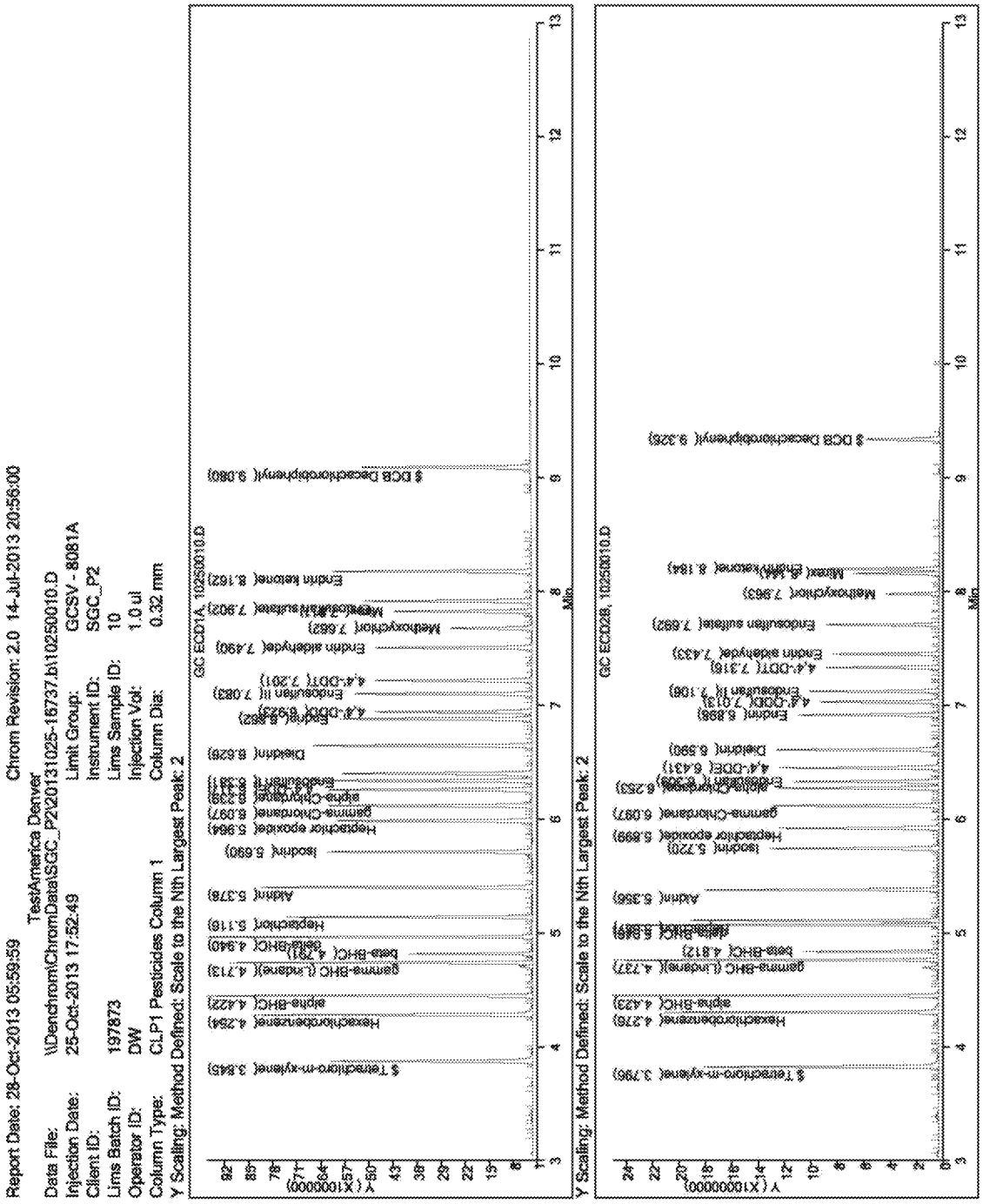
Table 8
Chrom Peak Number Assignment for Analytes
Columns CLPI and CLPII

Peak Number	Analyte
7	Tetrachloro-m-xylene
8	Hexachlorobenzene
9	Diallate
10	alpha-BHC
11	gamma-BHC (Lindane)
12	beta-BHC
13	delta-BHC
14	Chlordane (Technical)
15	Heptachlor
16	Aldrin
17	Chloropyrifos
18	Isodrin
19	Dicofol
20	Toxaphene
21	2,4'-DDE
22	Heptachlor Epoxide
23	gamma-Chlordane
24	alpha-Chlordane
25	4,4'-DDE
26	Endosulfan I
27	2,4'-DDD
28	Dieldrin
29	2,4'-DDT
30	Endrin
31	Kepone
32	4,4'-DDD
33	Chlorobenzilate
34	Endosulfan II
35	4,4'DDT
36	Endrin aldehyde
37	Methoxychlor
38	Mirex
39	Endosulfan sulfate
40	Endrin ketone
41	Propachlor
$41	Decachlorobiphenyl
42	Tris(2,3-dibromopropyl)phosphate

Table 9
Chrom Peak Number Assignment for Analytes
Columns RxiSil 35-MS & Rxi-XLB

Peak Number	Analyte
2 (IS)	1-Bromo-2-nitrobenzene
$3	Tetrachloro-m-xylene
4	Propachlor
5	Diallate
6	Hexachlorobenzene
7	alpha-BHC
8	gamma-BHC (Lindane)
9	Chlordane (Technical)
10	Technical Chlordane
11	beta-BHC
12	Heptachlor
13	delta-BHC
15	Aldrin
16	Chloropyrifos
17	Isodrin
18	Dicofol
19	Heptachlor Epoxide
20	gamma-Chlordane
21	2,4'-DDE
22	Toxaphene
23	alpha-Chlordane
24	Endosulfan I
25	4,4'-DDE
26	Dieldrin
27	2,4'-DDD
28	Chlorobenzilate
29	Endrin
30	2,4'-DDT
31	Kepone
32	4,4'-DDD
33	Endosulfan II
34	4,4'DDT
35	Endrin aldehyde
36	Endosulfan sulfate
37	Methoxychlor
38	Endrin ketone
39	Mirex
$40	Decachlorobiphenyl
41	Tris(2,3-dibromopropyl)phosphate

Attachment 1
Example Chromatograms – Columns CLPI & CLPII – AB Standard

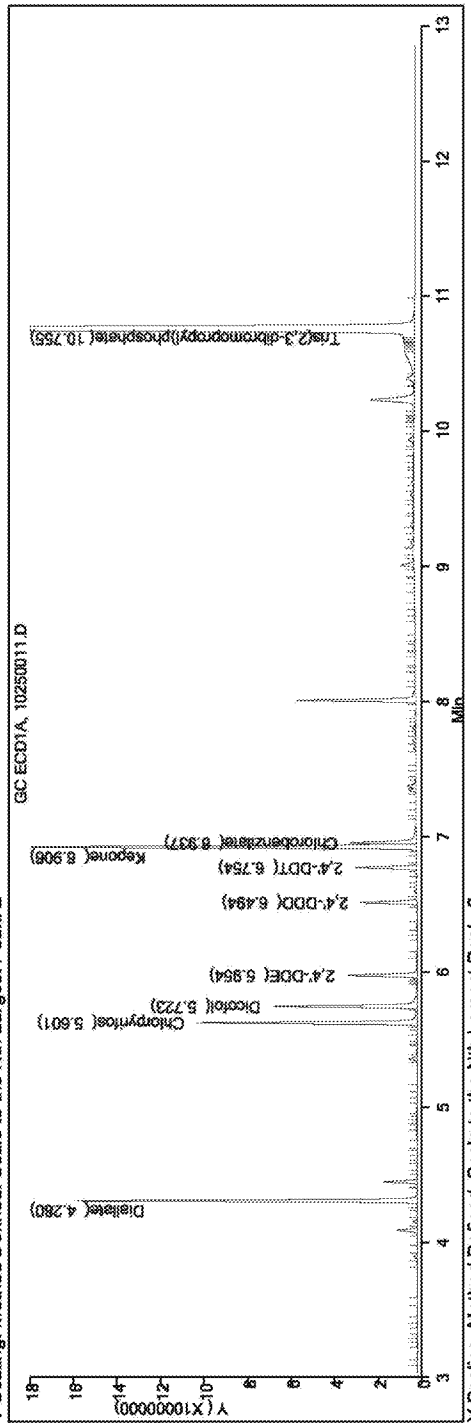


Attachment 2
Example Chromatogram – Columns CLPI & CLPII – AP9 Standard

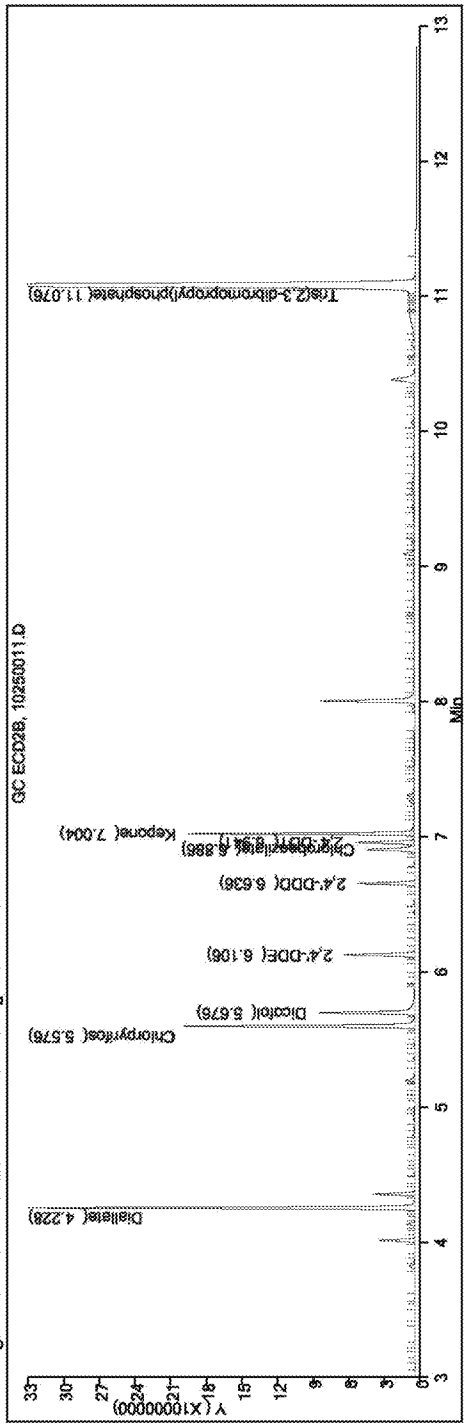
Report Date: 28-Oct-2013 05:59:59 Chrom Revision: 2.0 14-Jul-2013 20:56:00
TestAmerica Denver

Data File:	\\Denchrom\ChromData\SGC_P2\20131025-16737.b\10250011.D		
Injection Date:	25-Oct-2013 18:09:13	Limit Group:	GCSV - 8081A
Client ID:		Instrument ID:	SGC_P2
Lims Batch ID:	197873	Lims Sample ID:	11
Operator ID:	DW	Injection Vol:	1.0 ul
Column Type:	CLP1 Pesticides Column 1	Column Dia:	0.32 mm

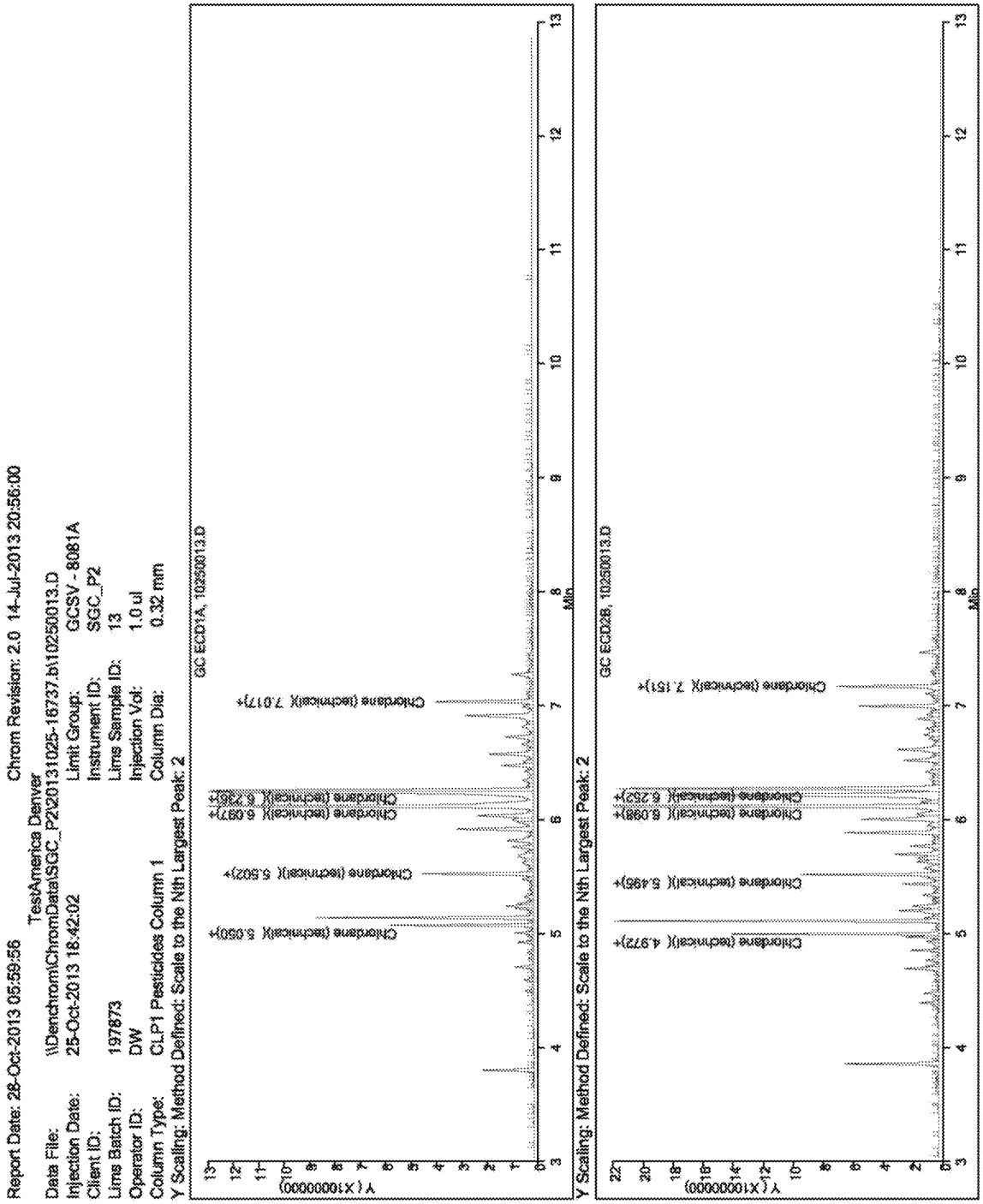
Y Scaling: Method Defined: Scale to the Nth Largest Peak: 2



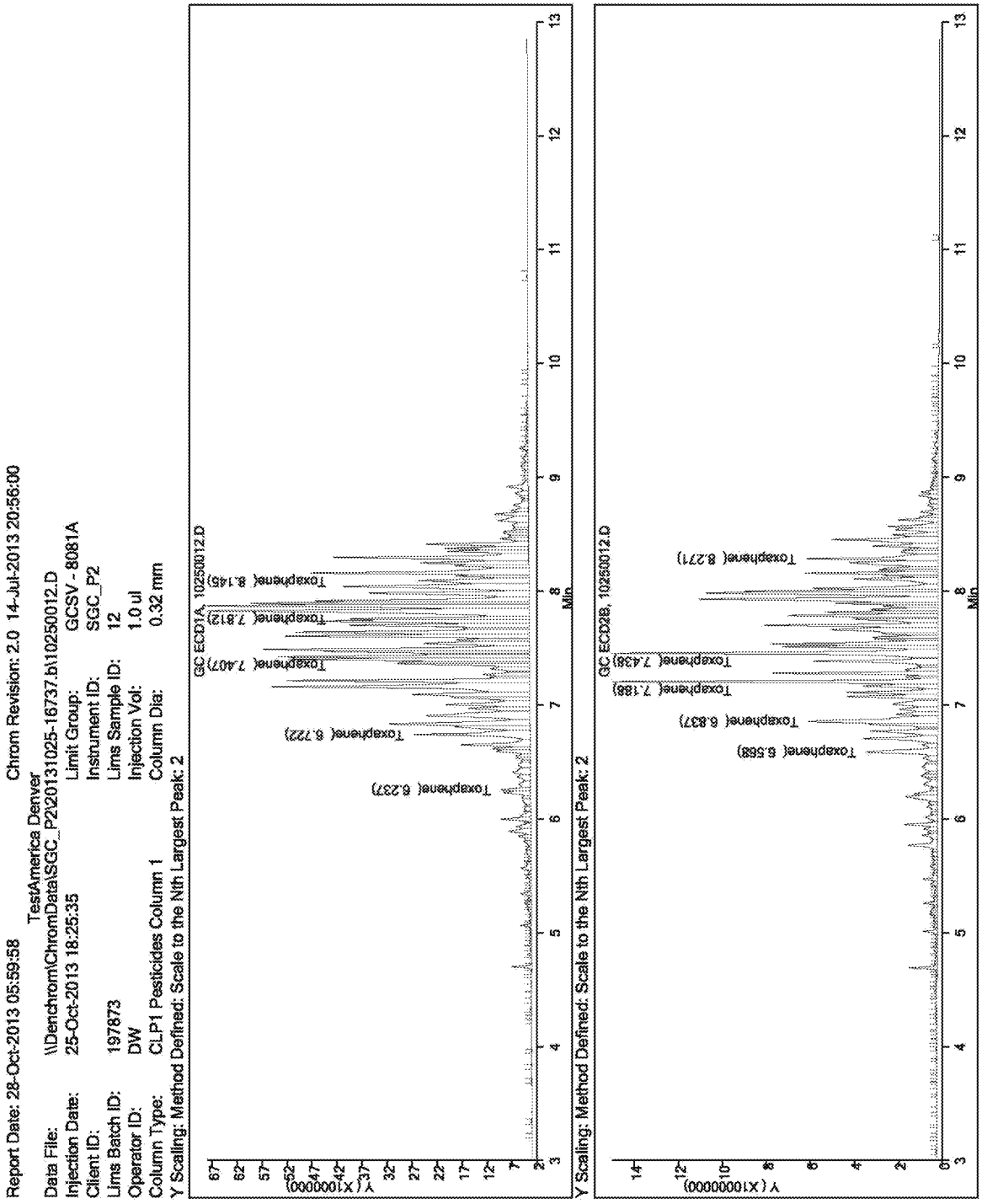
Y Scaling: Method Defined: Scale to the Nth Largest Peak: 2



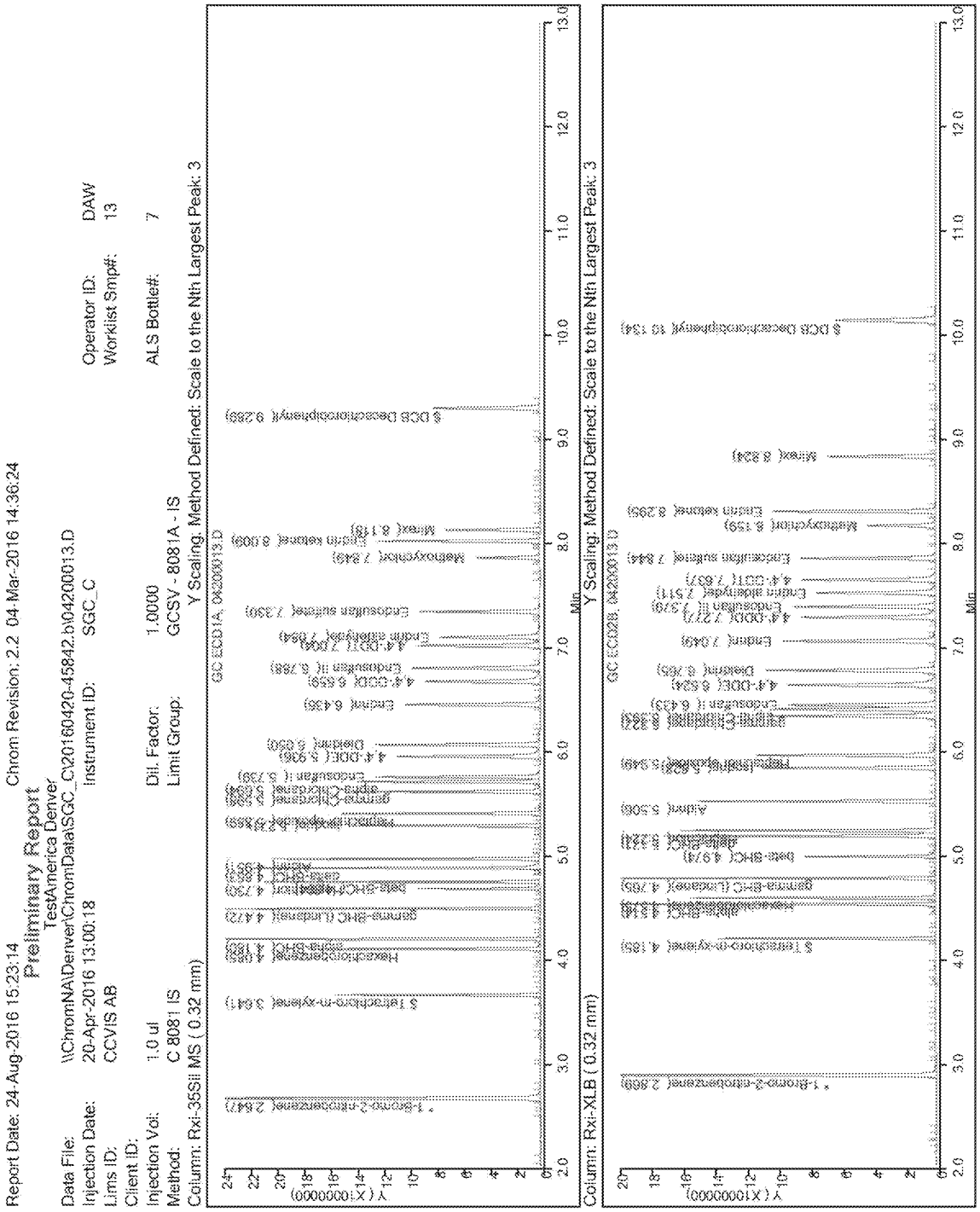
Attachment 3
Example Chromatogram – Columns CLPI & CLPII – Chlordane (Technical)



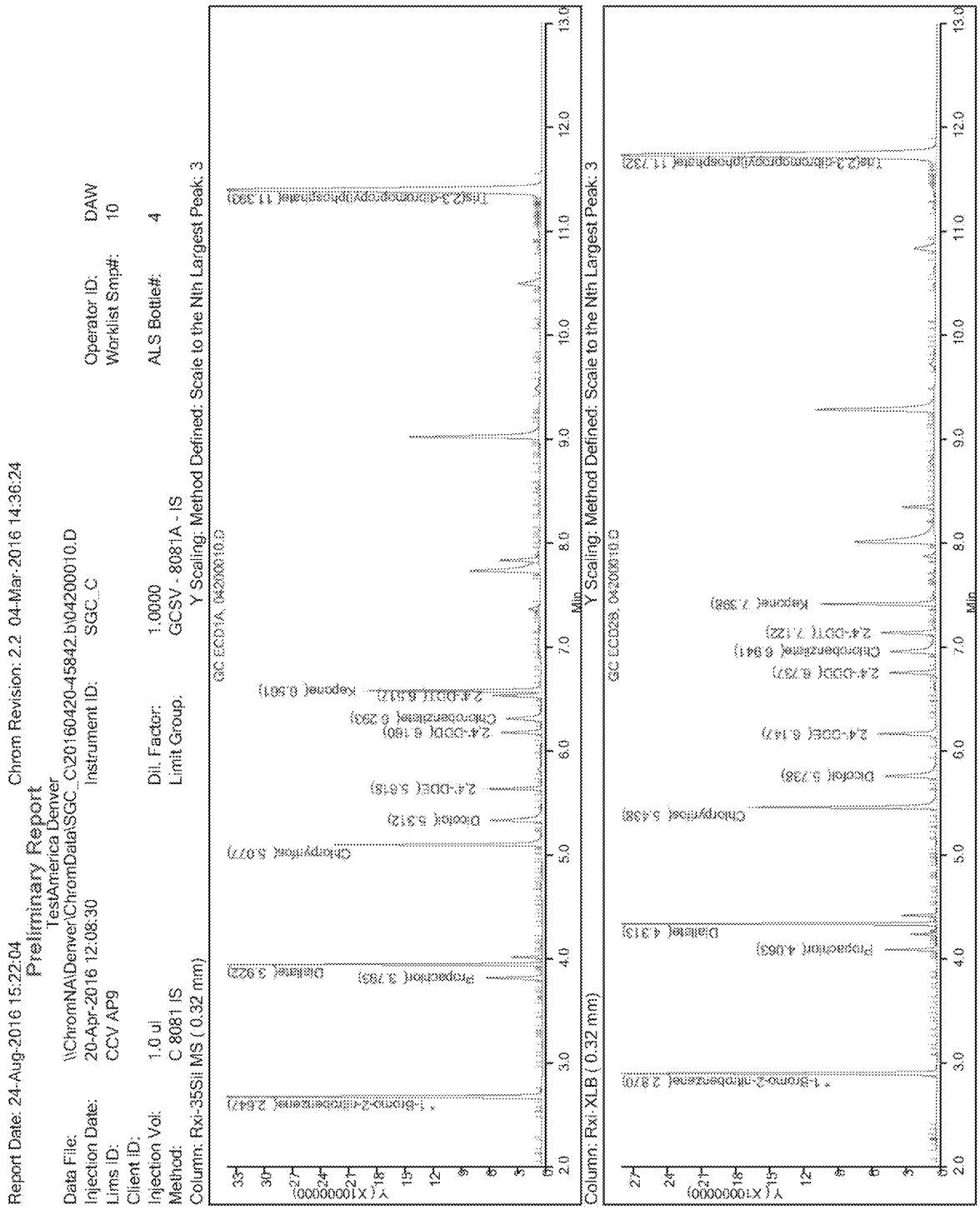
Attachment 4
Example Chromatogram – Columns CLPI & CLPII – Toxaphene



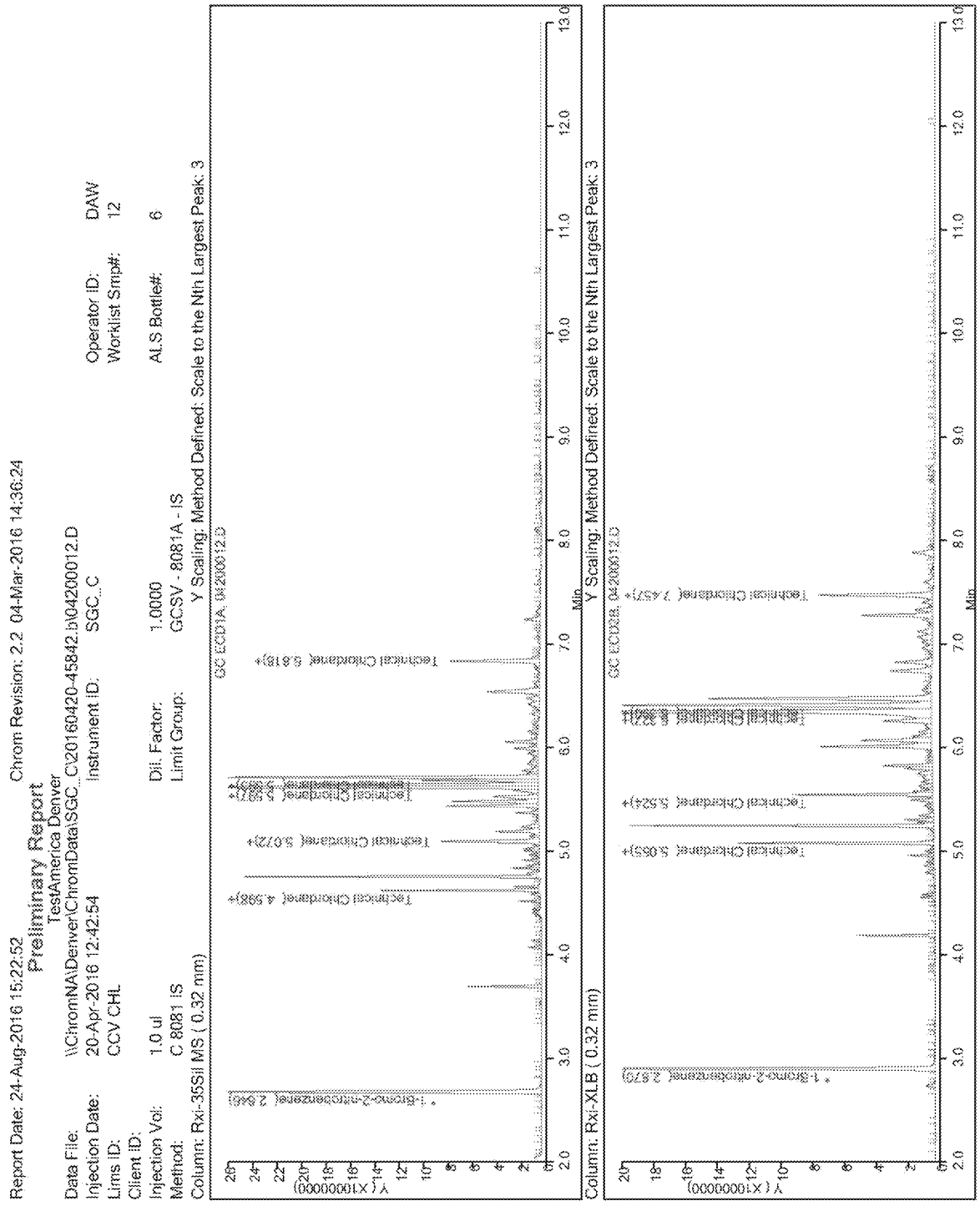
Attachment 5
Example Chromatograms – Columns RxiSil 35-MS & Rxi-XLB – AB Standard



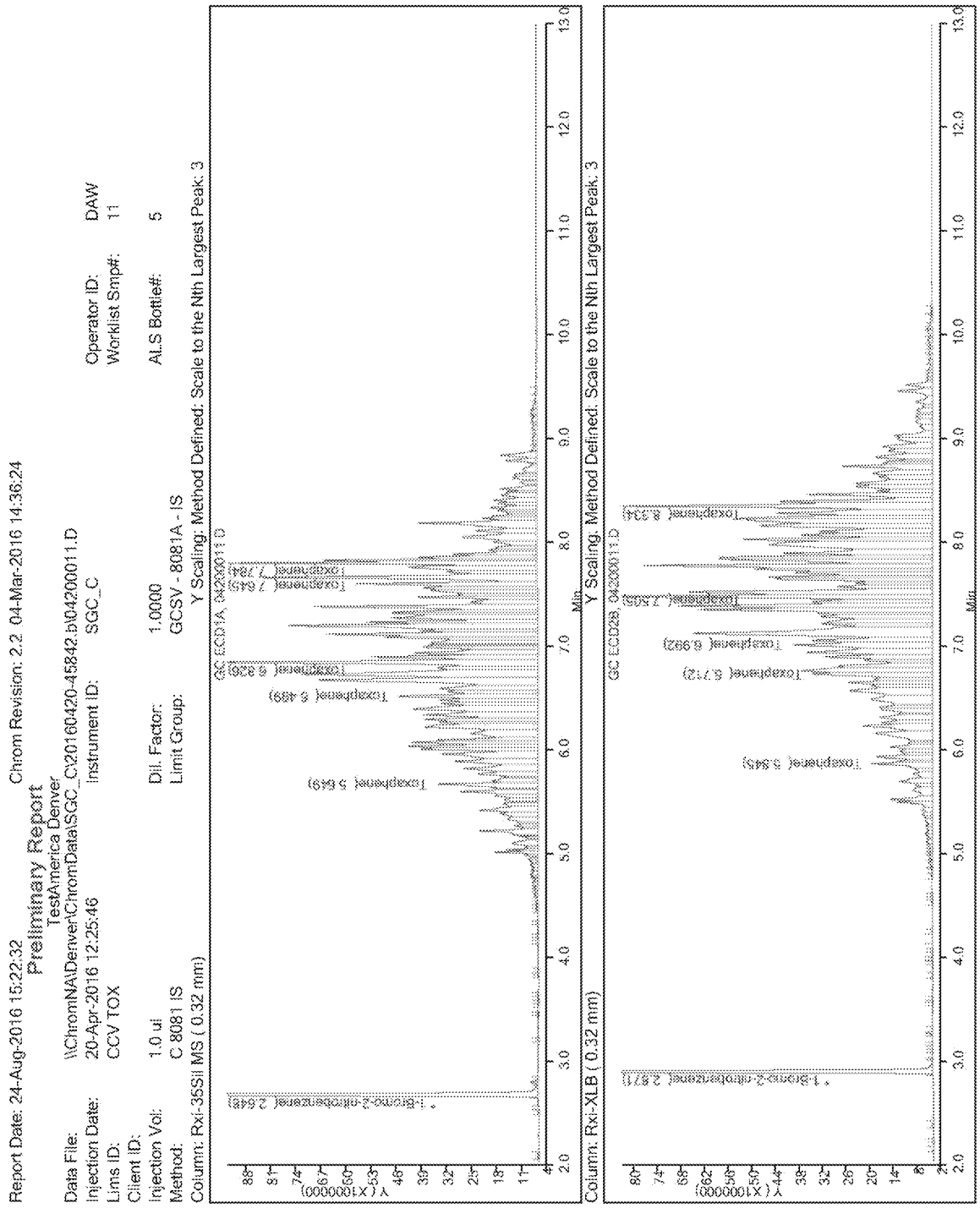
Attachment 6
Example Chromatograms – Columns RxiSil 35-MS & Rxi-XLB – AP9



Attachment 7
Example Chromatograms – Columns RxiSil 35-MS & Rxi-XLB
Chlordane (Technical)



Attachment 8
Example Chromatograms – Columns RxiSil 35-MS & Rxi-XLB – Toxaphene



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Title: Polychlorinated Biphenyls (PCBs) by GC/ECD [SW846 Methods 8082 and 8082A]

Approvals (Signature/Date):

2/28/20	2/28/20
Heather Fiedler — Date	~~Doug Gomer~~ RP 2/28/20 Read Pathroff — Date
Technical Specialist	Health & Safety Manager / Coordinator
3/2/20	3/2/20
Roxanne Sullivan — Date	Scott Hall — Date
Quality Assurance Manager	Laboratory Director

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1.0 Scope and Application

1.1 This SOP describes the procedure for the determination of concentrations of polychlorinated biphenyls (PCB) as Aroclors using the methodology prescribed in EPA SW-846 Method 8082 and 8082A.

1.2 This procedure is applicable to the gas chromatography analysis of extracts of aqueous, solid, and oil samples. When utilized for the analysis of oils, additional cleanup procedures may be required. This procedure also defines the conditions required when using a large volume injection.

1.3 This SOP does not include the procedures for extracting environmental samples. Refer to TestAmerica SOPs DV-OP-0006, DV-OP-0007, DV-OP-0015 and DV-OP-0016 for sample preparation procedures. Refer to SOP DV-OP-0012 for waste dilutions.

1.4 Additional information is provided in this SOP for the inclusion of the analysis of polychlorinated terphenyls (PCT) by the same protocols used for the determination of Aroclors.

1.5 This SOP does not include the determination of the concentration of PCB congeners.

1.6 Analytes, Matrix(s), and Reporting Limits

Tables 1 and LVI-1 list the specific Aroclors that are determined using this procedure and their associated reporting limits (RLs).

2.0 Summary of Method

2.1 Preparation

2.1.1 Aqueous Samples

PCBs are extracted from a one-liter aqueous sample with methylene chloride using a separatory funnel (SW-846 Method 3510). The extract is evaporated to approximately 25 mL and exchanged to hexane. The final extract volume is 10 mL, however depending on special client requirements the final extract volume can also be 1 mL or 5 mL. The extraction procedure is detailed in SOP DV-OP-0006.

2.1.2 LVI Aqueous Samples

PCBs are extracted from a 250 mL aqueous sample to a final volume of 5 mL with hexane (SW-846 Method 3510). The extraction procedure is detailed in SOP DV-OP-0006.

2.1.3 Solid Samples

PCBs are extracted from solid materials using either sonication or microwave extraction. If sonication extraction is selected the samples are

extracted with a 50:50 Acetone:Methlyene Chloride mixture, concentrated down to approximately 25 mL, exchanged with hexane, and brought to a 10 mL final volume. See DV-OP-0016 and DV-OP-0007 for details. If microwave extraction is selected the samples are extracted with a 50:50 Acetone:Hexane mixture, and concentrated down to a 10 mL final volume. See DV-OP-0015 and DV-OP-0007 for details.

2.1.4 Oil Samples

Oil samples are typically prepared by diluting 1 gram of sample to a final volume of 10 mL with hexane. The extraction procedure is detailed in SOP DV-OP-0012.

2.1.5 Wipe Samples

Wipes are typically collected using either filter paper or gauze. These samples can then be extracted using the procedure outlined in SOP DV-OP-0016.

2.1.6 Cleanup Procedures

Cleanup options are discussed in Section 4 below. Instructions for performing various cleanup procedures are detailed in SOP DV-OP-0007. All cleanups that are performed must be documented in an NCM.

2.2 Analysis

Samples are analyzed using a gas chromatograph with dual electron capture detectors (ECDs). Specific Aroclor mixtures are identified by the pattern of peaks compared to chromatograms of reference standards. The concentrations of Aroclors in the sample extract are determined using an internal standard calibration. Second column confirmation is only performed when requested by the client or as a program requirement. Work under the DoD/DOE QSM program and method 8082A require second column confirmation. The presence of multiple peaks that are characteristic of an aroclor in the sample serves as confirmation of analyte presence.

3.0 Definitions

3.1 Polychlorinated biphenyls (PCBs): PCBs are a class of organic compounds with 1 to 10 chlorine atoms attached to biphenyl, with a general chemical formula of $C_{12}H_{10-x}Cl_x$. There are 209 possible congeners.

3.2 Aroclor: PCBs were produced as technical mixtures by the chlorination of biphenyl. Production processes were designed to produce mixtures with characteristic chlorine contents. In the United States, most of the PCBs in the environment are in the form of Aroclors, which were produced by Monsanto from the 1930s through 1977. Each Aroclor mixture is identified by a four-digit number, the first two digits of which indicate the number of carbons in the biphenyl ring, i.e., 12, and the second two of which indicate the weight percent of chlorine. For example, Aroclor 1254 has

12 carbons and 54% by weight chlorine. The exception is Aroclor 1016, which has 12 carbons and 42% by weight chlorine.

NOTE: Each specific Aroclor produces a characteristic gas chromatographic pattern that represents the relative amounts of PCB congeners in the formulation. The formulation of the mixtures from batch to batch was fairly consistent, but never exactly the same. In almost all cases, the gas chromatogram can be used as a fingerprint to identify the specific Aroclor. Exceptions occurred for Aroclors 1254 and 1221. In each case, at least one batch was produced under different conditions, which resulted in an Aroclor mixture with the same approximate chlorine content, but with a significantly different distribution of congeners. These odd batches of 1254 and 1221 produce chromatographic patterns that are very different from the typical formulations. Standards for these odd batch Aroclors can be used to aid in the qualitative identification of Aroclors in environmental samples.

3.3 AR1660: Laboratory designation for the mixture of Aroclors 1016 and 1260.

3.4 AR2154: Laboratory designation for the mixture of Aroclors 1221 and 1254.

3.5 AR3262: Laboratory designation for the mixture of Aroclors 1232 and 1262.

3.6 AR4268: Laboratory designation for the mixture of Aroclors 1242 and 1268.

3.7 Polychlorinated Terphenyls: Polychlorinated terphenyls (PCTs) are chemically related to PCBs with the exception that PCTs have an additional phenyl group. The PCTs included in this analysis are AR 5432, AR 5442, and AR 5460. The preparation and analysis is treated the same as for the PCB Aroclor analysis.

3.8 Lower Limit of Quantitation (LLOQ): The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be $\geq$ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

4.0 Interferences

4.1 Hydrocarbons can co-elute and thereby mask the Aroclor pattern. The laboratory uses acid cleanup with concentrated sulfuric acid to remove hydrocarbons from solid and oil sample extracts, and for water samples when extracts have noticeable color or whenever there is clear evidence of interferences in the initial sample chromatograms. Acid cleanup removes low-to-medium molecular weight polar organic interferences from sample extracts. Detailed instructions for performing acid cleanup are provided in SOP DV-OP-0007.

All QC is brought through the cleanup process and reported with the samples. An aliquot of all samples and QC is set aside and not brought through the cleanup process. If the QC is out of criteria due to the cleanup process then the QC that

wasn't brought through the cleanup process will be analyzed and used to verify the batch for the samples not brought through clean-up.

4.2 Sulfur will interfere and can be removed using procedures described in SOP DV-OP-0007.

4.3 Contamination by carryover can occur when a low concentration sample is analyzed after a high concentration sample. Generally any sample that has a concentration greater than the highest calibration point will warrant consideration for carryover. Any affected samples are re-analyzed.

4.4 Interferences in the GC analysis arise from many compounds amenable to gas chromatography that give a measurable response on the electron capture detector.

4.4.1 Phthalate esters, which are common plasticizers, can pose a major problem in the determinations. Interferences from phthalates are minimized by avoiding contact with any plastic materials.

4.4.2 Single-component chlorinated pesticides, if present, may co-elute with individual PCB congeners and interfere with the identification and/or quantitation of the aroclors. This can be addressed by analyzing a chlorinated pesticide mixed standard prior to an initial calibration to identify where potential interferences might occur.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (M-E-001 DV), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.2.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2.4 All ^{63}Ni sources shall be leak tested every six months, or in accordance with the manufacturer's general radioactive material license. All ^{63}Ni sources shall be inventoried every six months. If a detector is missing, the TestAmerica Denver Radiation Safety Officer and the TestAmerica Corporate EH&S Director shall be immediately notified and a letter sent to the Colorado Department of Public Health and Environment. Always use proper procedures for the safe handling of radioactive materials when working on the GC parts that come into direct contact with the ECD.

5.3 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Serious or Significant Hazard Rating

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Acetone	Flammable	1000 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm (TWA)	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm (TWA) 125 ppm (STEL)	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Hydrogen gas	Explosive	None	The main hazard is flammability. Exposure to moderate concentrations may cause dizziness, headache, nausea, and unconsciousness. Exposures to atmospheres less than 8 to 10% oxygen will bring about sudden unconsciousness, leaving individuals unable to protect themselves. Lack of sufficient oxygen may cause serious injury or death.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Sulfuric Acid	Corrosive Carcinogen	1 mg/m3	Inhalation may cause irritation of the respiratory tract with burning pain of the nose and throat, coughing, wheezing, shortness of breath, and pulmonary edema. Causes chemical burns to the respiratory tract. Inhalation may be fatal as a result of spasm, inflammation, edema of the larynx and bronchi, chemical pneumonitis, and pulmonary edema. Causes skin burns. Causes severe eye burns. May cause irreversible eye injury, blindness, permanent corneal opacification.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

A gas chromatographic system with dual columns and dual ECD (^{63}Ni) detectors, and a data system capable of measuring peak area and/or height. The current instruments that are in use for this analysis are HP6890N-instrument P3 and HP5890 Series II- instrument W. Instrument R- HP5890 Series II may be used on occasion.

6.2 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Columns

6.3.1 Primary Column: Rtx CLP Pest, 30 m x 0.32 mm id, 0.5 µm coating.

6.3.2 Secondary Column: Rtx CLP Pest II, 30 m x 0.32 mm id, 0.25 µm coating.

6.3.3 Additional columns that can be used for confirmation include 30m x 0.32mm id HP-5 or HP-1701.

6.4 Supplies

6.4.1 Autosampler vials, crimp caps with PTFE-faced septa.

6.4.2 Y-splitter, septa, guard columns, ferrules, Agilent deactivated injection port liners, Siltek glass wool.

6.4.3 Microsyringes, various sizes, for standards preparation, sample injection, and extract dilution.

6.4.4 Various class A volumetric flasks from 5 mL to 250 mL.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Acetone, 99.4% for organic residue analysis. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.2 Hexane, pesticide grade. Each lot is tested for purity prior to use per SOP S-T-001.

7.1.3 Carrier Gas: ≥ 99.99999% pure hydrogen

7.1.4 Make-up Gas: ≥ 99.99980% pure nitrogen

7.2 Stock Standards

7.2.1 All standards are subject to verification using a second-source standard before they are used for sample analysis. This process is described in SOP DV-QA-0015.

7.2.2 All standards must be refrigerated at 0-6 ºC. All stock standards must be protected from light. Stock standard solutions should be brought to room temperature before use.

7.2.3 Stock standards are monitored for signs of degradation or evaporation. The standards must be replaced annually from the date of opening or earlier if the vendor indicates an earlier date.

7.2.4 Dilutions from stock standards cannot have a later expiration date than the date assigned to the parent stock solutions. The standards must be replaced at least every six months, or sooner if comparison with check standards indicates a problem.

7.3 PCB and Surrogate Stock Calibration Standards

7.3.1 Stock A (AR_(*Aroclor #*)_RES

For each of the Aroclors listed in Tables 1 and LVI-1, a commercially prepared stock standard solution is obtained. Each stock standard contains the specific Aroclor in pesticide-grade hexane (or in some cases, isooctane) at a concentration of 1,000 µg/mL. The current vendor is RESTEK (PCB/Catalog#) AR1016/32006, AR1221/32007, AR1232/32008, AR1242/32009, AR1248/32010, AR1254/32011, AR1260/32012, AR1262/32409, and AR1268/32410, other vendors may be used.

Surrogate Stock B (AR_SURR_RES)

A commercially prepared stock standard solution is obtained that contains the surrogate compounds tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) in acetone, at a concentration of 200 µg/mL. The current vendor is RESTEK catalog #32000, other vendors may be used.

Internal Standard Stock

A commercially prepared stock standard solution is obtained that contains the internal standard 1-bromo-2-nitrobenzene in acetone, at a concentration of 1000 µg/mL. The current vendor is RESTEK catalog #32279, other vendors may be used.

7.3.2 PCT Stock

A commercially prepared stock standard solution is obtained that contains the individual PCT compounds at a concentration of 35 ug/mL in hexane. The current vendor is Accustandard and the catalog numbers are AR 5432 T432S, AR 5442 T442S, and AR 5460 T460S. Equivalent standards from other vendors and at other concentrations may be used.

7.4 Intermediate and Working Level Calibration Standard Solutions

7.4.1 Stock C (Level 7 Calibration) Standard Solutions

(AR_*(aroclor #)*_L*(level)*

A Stock C standard solution is prepared for the various Aroclors or combination of Aroclors as summarized in the following table. In each case, the Stock C standard solution is also the highest concentration (i.e., Level 7) calibration standard.

Stock C (Level 7)	Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.1 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	1.0
	0.1 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	1.0
	0.025 mL of surrogate Stock B	200		TCMX	0.05
				DCB	0.05
AR_2154	0.1 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	1.0
	0.1 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	1.0
AR_3262	0.1 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	1.0
	0.1 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	1.0
AR_4268	0.1 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	1.0
	0.1 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	1.0
AR_1248	0.1 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	1.0

ARHVI_*(aroclor #)*_L*(level)*

Stock C (Level 7)	LVI Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
AR_1660	0.05 mL of Aroclor 1016 Stock A	1000	100	Aroclor 1016	0.5

Stock C (Level 7)	LVI Recipe	Conc (µg/mL)	Final Vol (mL)	Final Concentrations (µg/mL)	
	0.05 mL of Aroclor 1260 Stock A	1000		Aroclor 1260	0.5
	0.0125 mL of surrogate Stock B	200		TCMX	0.025
				DCB	0.025
AR_2154	0.05 mL of Aroclor 1221 Stock A	1000	100	Aroclor 1221	0.5
	0.05 mL of Aroclor 1254 Stock A	1000		Aroclor 1254	0.5
AR_3262	0.05 mL of Aroclor 1232 Stock A	1000	100	Aroclor 1232	0.5
	0.05 mL of Aroclor 1262 Stock A	1000		Aroclor 1262	0.5
AR_4268	0.05 mL of Aroclor 1242 Stock A	1000	100	Aroclor 1242	0.5
	0.05 mL of Aroclor 1268 Stock A	1000		Aroclor 1268	0.5
AR_1248	0.05 mL of Aroclor 1248 Stock A	1000	100	Aroclor 1248	0.5

7.4.2 AR_1660 Calibration Levels

A total of 7 calibration standards are prepared for AR_1660 as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the Aroclors, but only the AR_1660 calibration standards include the surrogates. In all cases, measured volumes of the Stock C standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Stock C Used (mL)	Final Volume (mL)	Final PCB Conc (µg/mL)	Final Surrogate Conc (µg/mL)*
1	0.25	10	0.025	0.00125
2	0.5	10	0.050	0.0025
3	1.0	10	0.10	0.005
4	2.5	10	0.25	0.0125
5 (CCV)	5.0	10	0.50	0.025
5 (CCV) 1660 mix only	12.5	25	0.5	0.025
6	7.5	10	0.75	0.0375
7 (Stock C)	--	--	1.0	0.0500
* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				
LVI Level	**Vol of LVI Stock C Used (mL)**	**Final Volume (mL)**	**Final PCB Conc (µg/mL)**	**Final Surrogate Conc (µg/mL)***
1	0.25	10	0.0125	0.00063
2	0.5	10	0.025	0.00125
3	1.0	10	0.05	0.0025
4	2.5	10	0.125	0.0625

5 (CCV)	5.0	10	0.25	0.0125
5 (CCV) 1660 mix only	12.5	25	0.25	0.0125
6	7.5	10	0.375	0.0188
7 (Stock C)	--	--	0.50	0.0250
* Surrogates are in the AR_1660 calibration solutions only. None of the other Aroclor calibration solutions contain the surrogate compounds.				

7.4.3 Working Single-Point PCB Calibration Standards

The Level 5 standard in the table above is used for single-point calibrations of the individual Aroclors. These standards are also used as pattern recognition standards.

7.4.4 Polychlorinated Terphenyl Calibration Levels

A total of 7 calibration standards are prepared for PCTs as summarized in the following table. As needed, the following table can be used to prepare calibration standards for any of the PCTs. The level 7 standard is prepared from the stocks described in section 7.3.3 by diluting 1 mL of the stock to 35 mL final volume with hexane. The final concentration of the level 7 standard is 1.0 ug/mL. In all cases, measured volumes of the Level 7 standard are diluted using pesticide-grade hexane to the final volume indicated in the following table.

Level	Vol of Level 7 Used (mL)	Final Volume (mL)	Final PCT Conc (µg/mL)
1	0.25	10	0.025
2	0.5	10	0.05
3	1	10	0.10
4	2.5	10	0.25
5 (CCV)	5	10	0.50
6	7.5	10	0.75

7.5 Second-Source Standards for Initial Calibration Verification (ICV)

These standards are purchased from a vendor different from the one that supplied the stock calibration standards.

7.5.1 Second-Source Stock A' Aroclor Standard Solutions (AR_*(aroclor#)*_RESss

Commercially prepared solutions in pesticide-grade hexane (or isooctane) are routinely obtained for Aroclors 1016 and 1260. The Aroclor concentration in each solution is 1000 µg/mL. A second source may be obtained for the other Aroclors, if necessary. The current second source is RESTEK (PCB/Catalog#) AR1221/32007.sec; AR1016/1260/32039.sec; AR1232/32008.sec; AR1242/32009.sec; AR1248/32010.sec;

AR1254/32011.sec; AR1262/32409.sec; AR1268/32410.sec, other vendors may be used.

Second source surrogate

A commercially prepared solution is obtained containing TCMX and DCB each at a concentration of 200 ug/mL. The current second source surrogate is Restek 32000.

Working Level ICV AR_*(aroclor#)*_ICV

The working level second source ICV standard is prepared by combining 0.025 mL of Aroclor 1016 Stock A', 0.025 mL of Aroclor 1260 stock A', and 0.00625 mL of the surrogate second source stock and diluting to a final volume of 100mL with pesticide grade hexane. This results in a concentration of 0.25 ug/mL for each of the Aroclors and 0.0125 ug/mL for each surrogate. If a second source verification standard is prepared for any of the Aroclors other than the AR_1660 mixture, the surrogates are not added to that mix.

7.5.2 PCT Second Source Stock and working level.

7.5.2.1 A commercially prepared solution of each of the PCT mixes is obtained from a different vendor and typically prepared at a concentration of 100 ug/mL in hexane. The current second source vendors and catalog numbers are AR 5432 Chem Services F290RPS, AR 5442 Chem Services F860RPS, and AR 5460 Chem Services F292RPS. Other vendors may be used.

7.5.2.2 The working level PCT standard is prepared at a concentration of 0.25 ug/mL by diluting 0.025 mL of each stock to a final volume of 10 mL.

7.6 Continuing Calibration Verification Standard (CCV), 0.5 µg/mL

The working CCV solution is the same as the Level 5 initial calibration standard, as shown in the table in Section 7.4.2.

7.7 RL Standard

The lowest concentration calibration standard (i.e., Level 1) is used as the RL Standard.

7.8 Laboratory Control Standard (LCS) Spiking Solution (AR1660)

NOTE: The LCS/MS spiking solution (8082LCS) is prepared and used as part of the scope of the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015, and DV-OP-0016. The following information is provided for reference only.

In general the LCS is made up at a concentration of 2 µg/mL in a water-soluble solvent such as acetone. For oil samples extracted by waste-dilution, the standard

is made in hexane. The standard contains Aroclors 1016 and 1260 only. Typically 1 mL of this standard is added to 1 liter of water samples, 30 g of soil samples, or 1 g of oil samples. The current LCS vendor is Restek at a concentration of 1000ug/ml. The solution is prepared by diluting 0.5 ml of this stock into 250 ml with acetone solvent.

7.9 Matrix Spike (MS) Spiking Solution:

The working matrix spike solution is the same as the LCS spike solution. Matrix spike samples are prepared by adding 1.0 mL of the working solution to a second one-liter aliquot of the selected aqueous sample, or to a 30-gram subsample of the selected soil sample. The MS duplicate (MSD) is prepared in the same way using a third aliquot of the selected sample.

7.10 Surrogate Spike Solution

7.10.1 Stock Surrogate Spike Solution:

A commercially prepared solution containing 200 µg/mL each of decachlorobiphenyl (DCB) and tetrachloro-m-xylene (TCMX) in acetone is purchased. The current source is Restek 32000.

7.10.2 Working Surrogate Spike Solution (8081/82 Surr)

NOTE: Samples are spiked with the surrogate compounds during sample preparation, which is described in the organic preparation SOPs DV-OP-0006, DV-OP-0012, DV-OP-0015 and DV-OP-0016. The following information is provided for reference only.

The working level surrogate solution is made up to contain DCB and TCMX at a concentration of 0.2 µg/mL. For water and soil samples the solution is made in a water-soluble solvent like acetone. For all oil samples extracted by waste dilution the solution is made in hexane.

7.11 Internal Standard Spiking solution 1 µg/mL (BNB IS)

An intermediate stock (BNB stock) at 10 µg/mL is prepared by diluting 1 mL of the commercial Internal Standard Stock from Section 7.3 to a final volume of 100 mL in hexane. The Internal standard spiking solution is then prepared by diluting 10 mL of this intermediate to a final volume of 100 mL. Every standard, QC sample, and client sample is spiked with 0.1 mL of the internal standard spiking solution into 1 mL. For the LVI preparation 0.05 mL of internal standard is spiked into 1 mL of sample or standard.

7.12 Primer Mix

The primer mix typically consists of a mixture of CCV standards and/or old calibration standards. The concentrations of the components of the primer mix are not critical. The primer mix is injected one or more times prior to analyzing standards and samples to ensure that the chromatographic system is stable, i.e., that retention times are reproducible.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[2]	Reference
Water[1]	Amber glass	1 Liter	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846
Water[3]	Amber glass	250 mL	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846
Solid	Glass	8 oz	Cool, ≤ 6°C	1 Year to extraction 40 days to analysis	SW-846

[1]To achieve routine reporting limits, a full one liter of sample is required. Additional one-liter portions are needed to satisfy the requirements for matrix spikes and duplicate matrix spikes.

[2]California, Connecticut, Pennsylvania and South Carolina do not allow the 1 year holding time. For work performed in these states, the extraction holding time is 7 days for water and 14 days for solid.

[3]Samples collected in 250 mL bottles will be extracted by SW846 method 3510 followed by analysis using the LVI procedure described in this SOP.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD/DOE QSM 5.0or 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.0 or 5.1 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as

appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Performance Studies

Before analyzing samples, the laboratory must establish a method detection limit (MDL). The current MDL value is maintained in the TestAmerica Denver LIMS. In addition, an initial demonstration of capability (IDOC) must be performed by each analyst on an instrument he/she will be using. On-going proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Batch Definition

Batches are defined at the sample preparation stage. A batch is a set of up to 20 samples of the same matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. The minimum batch QC in each run is an acceptable method blank or instrument/calibration blank. See QA Policy DV-QA-003P for further details. For DoD QSM 4.2 or DOD/DOE QSM 5.0 or 5.1, the MS/MSD must be from the project site and if insufficient sample to analyze the MS/MSD pair is available, this is documented in an NCM and an LCSD is performed.

9.4 Method blank

A method blank is prepared and analyzed with each batch of samples. The method blank consists of reagent water (for aqueous sample batches) or Ottawa sand (for solid sample batches) to which the surrogate compounds are added. The method blank is subject to the entire extraction and analysis process. The method blank must be analyzed on every instrument that is used to analyze the samples from the batch.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (RL) or above one-tenth of the concentration found in the associated samples. See section 9.7 for further evaluation of the surrogate in the MB.

Corrective Action: If the method blank exceeds allowable levels, the source of the contamination must be investigated and all associated samples that produced detections for the contaminant must be re-extracted and reanalyzed. Any samples that produce concentrations more than 10 times the concentration of the same compound as the blank contaminant may be reported with proper flagging and narration. Method Blanks for South Carolina compliance work MUST be below the RL.

9.5 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed with each batch of samples. The LCS is prepared as described in Section 7.8. The LCS is subject to the entire extraction and analysis process.

Acceptance Criteria: The LCS recovery must be within the established control limits. The laboratory's standard control limits are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise. Current control limits are maintained in LIMS. South Carolina requires the LCS to recover within limits of 70-130%.

Corrective Action: If recoveries are not within the established limits, the analytical system is out of control and corrective action must occur. All associated samples must be re-extracted and reanalyzed. If the LCS exceeds the upper control limit then all samples that do not contain detections for the affected compound may be reportable with client consent and proper flagging and narration. Note that exceptions for an out of control LCS are not allowed for DoD/DOE QSM 5.0 or 5.1 projects unless prior approval is indicated in the projects instructions.

9.6 Matrix Spike (MS) and Matrix Spike Duplicate Samples (MSD)

One MS/MSD pair is required with each analytical batch. Note that some programs (e.g., North Carolina and South Carolina) require preparation and analysis of an MS/MSD pair at a 10% frequency. Preparation of the MS is described in Section 7.9. The MSD is another aliquot of the sample selected for the MS that is spiked in the same manner as the MS. For DoD QSM 4.2 or DOD/DOE QSM 5.0 or 5.1, the MS/MSD must be from the project site and if insufficient sample to analyze the MS/MSD pair is available, this is documented in an NCM but no LCSD is performed.

Acceptance Criteria: The MS and MSD recoveries and the relative percent difference (RPD) between the MS and MSD results must be within the established control limits. Percent recovery control limits are set at ± 3 standard deviations around the historical mean of the LCS recovery data, unless otherwise dictated by the client or project. The RPD control limit is set at 3 standard deviations above the mean of the historical data.

NOTE: DOD/DOE QSM 5 limits apply to projects performed under this program.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in

order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.7 Surrogates

Each field sample, QC sample, and each calibration standard that is used for the AR_1660 initial calibration, is spiked with surrogate compounds decachlorobiphenyl (DCB) and trichloro-m-xylene (TCMX). The surrogate spike solution is prepared as described in Section 7.10.

Acceptance Criteria: The surrogate recoveries must be within the established control limits, which are set at ± 3 standard deviations around the historical mean, unless project requirements dictate otherwise.

Corrective Action: If recoveries of the surrogates in blanks are outside of the control limits, check for calculation or instrument problems. High recoveries might be acceptable if the surrogate recoveries for the samples and other QC in the batch are acceptable. Low surrogate recoveries in the blank require re-extraction and reanalysis of the associated samples especially those that have detections for the targeted compounds that are found in the blank. Samples that are ND and have the surrogates in control may be reportable with proper flagging and NCM.

For field samples, surrogate recovery is calculated and reported for DCB only. TCMX may also be added. However, if both surrogate compounds are added, and recoveries calculated, and either surrogate fails to fall within the control limits, corrective actions are required (this also applies to programs that require the use of only one surrogate). Samples with surrogate recoveries that are above the upper control limit may be reportable with flagging and narration if they do not have reportable detections. See SOP DV-QA-003P for further evaluation.

If matrix interference is not obvious from the initial analysis, it is only necessary to re-extract and reanalyze a sample once to demonstrate that poor surrogate recovery

is due to matrix effects, as long as the extraction/instrument system is proven to be working properly.

9.8 Internal standard

Acceptance Criteria: The internal standard recoveries must be within -50% to 200% for standard solutions of the recovery established by the midpoint of the ICAL or the opening CCV for the run on days when an ICAL is not analyzed.

DOD/DOE QSM 5.0 does not have criteria for internal standards. The above criteria is used. DOD/DOE QSM 5.1 uses the above criteria.

Corrective Action: If the internal standard response is outside of this range then the samples must be diluted until the recoveries are in control. Failure to meet this criteria in a CCV requires reanalysis of the standard and all affected samples analyzed in the bracket previous to the standard and after the standard. Recalibration is necessary if control cannot be established. For DOD/DOE QSM 5.1, if corrective action fails in field samples, the data must be qualified and explained in the case narrative. Flagging is not appropriate for failed standards.

10.0 Calibration and Standardization

10.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.1 Use the ChemStation chromatography data system to set up GC conditions for calibration. See Tables 2 and LVI-2 for typical operating conditions. The conditions described in Table LVI-2 are to be used when performing the large volume injection approach.

10.1.2 Transfer calibration standard solutions into autosampler vials and load into the GC autosampler. Use the Chrom worklist to set up the analytical sequence.

10.1.3 Transfer unprocessed calibration data to the Chrom database for processing. After processing the calibration data, print the calibration report and review it using the calibration review checklist (GC and HPLC Data Review Checklist - ICAL). Submit the calibration report to a qualified peer or the group leader for final review. The completed calibration checklist is scanned and stored in the documents section of each analytical batch.

10.2 A new calibration curve must be generated initially, after major changes to the system, or when continuing calibration criteria cannot be met. Major changes include installation of new columns.

10.3 Initial Calibration (ICAL)

10.3.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003 *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

10.3.2 An internal standard calibration using seven concentration levels of the AR_1660 mixture is routinely performed. (At least five calibration levels are required.) This provides concentration levels for Aroclor 1016, Aroclor 1260, and the surrogate compounds DCB and TCMX.

10.3.2.1 Prior to the analysis of the ICAL the resolution for the triplet towards the end of the Aroclor 1260 chromatogram for peaks 1&2 and peaks 2&3 must be <75% on one of the two columns used for the analysis.

10.3.2.2 See Tables 3 and LVI-3 for Calibration Levels. Calibration levels defined in Table LVI-3 are appropriate when the large volume injection approach is used.

10.3.2.3 The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes. This run is <u>not</u> part of the actual calibration.

10.3.2.4 Prior to analysis of the initial calibration standards it is recommended that a chlorinated pesticide standard (Method 8081) be analyzed as a locater standard to identify potential interferences in samples due to the presence of chlorinated pesticides.

10.3.3 All initial calibration points must be analyzed without any changes to instrument conditions, and all points must be analyzed within 24 hours.

10.3.4 The calibration curves for Aroclors 1016 and 1260 and the surrogate compounds are modeled either as average response factors or as calibration curves using a systematic approach to selecting the optimum calibration function.

10.3.5 The calibration for each of the other Aroclors (see Table 1 or LVI-1) is initially determined using a single, mid-level calibration standard. As needed, the laboratory may generate a multi-point calibration for other commonly detected Aroclors, such as 1221, 1254, and 1248. When additional multi-point calibrations are developed for the other Aroclors, a second-source ICV standard is also analyzed. When Aroclors are detected, they must be re-analyzed after an acceptable calibration curve (minimum five points) and its verification (ICV) are obtained.

NOTE: Samples from sites known to be contaminated with specific Aroclors should be analyzed using a multi-point calibration curve

for the identified Aroclors. This information is provided to the analyst through special instructions in LIMS.

NOTE: Generally, it is NOT acceptable to remove points from a calibration for the purposes of meeting calibration criteria. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of the preparation of the calibration standards. Any problems found must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed

10.3.6 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious mis-injection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

10.3.6.1 The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and

10.3.6.2 The lowest remaining calibration point is still at or below the project reporting limit; and

10.3.6.3 The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and

10.3.6.4 The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

10.3.6.5 If a data point is rejected, it must be documented on the calibration checklist, in the sequence log and on an NCM which is filed with each project that is reported from the calibration.

10.3.7 The high and low standard for the initial calibration of the AR_1660 mixture defines the acceptable quantitation range for all of the Aroclors. The low calibration standard must be at or below the RL. If a sample extract contains any Aroclor at a concentration that exceeds the upper range of the calibration, then the extract must be diluted with hexane and reanalyzed.

10.3.8 Select 5 major peaks in the analyte pattern (only 3 peaks are usable for Aroclor 1221). The peaks that are chosen should have responses that are at least 25% of the response for the largest peak in the Aroclor pattern, with the exception of Aroclor 1268 where the requirement is 10%. Try to include one peak that is unique (differs in size or location relative to the other common Aroclors) to the Aroclor being quantitated. Calculate the response of each of the major peaks for each Aroclor, and use these responses independently, averaging the resultant concentrations found in

samples for a final concentration result. When using this option, it is appropriate to remove peaks that appear to be co-eluting with contaminant peaks from the quantitation (i.e., peaks that are significantly larger than would be expected from the rest of the pattern).

NOTE: A minimum of three accurate peaks must be used to quantify an Aroclor (two for Aroclor 1221).

10.4 Internal Standard Calibration

Internal standard calibration involves the comparison of an instrument response (e.g., peak area or peak height) from the target compound in the sample to the response of the internal standard compound, which is added to the sample or sample extract prior to injection. The same concentration of internal standard is added to each initial calibration standard. For each calibration level, the response factor, RF, is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$ *Equation 1*

Where:

A_s = Peak area (or height) of the analyte or surrogate.
A_{is} = Peak area (or height) of the internal standard.
C_s = Concentration of the analyte or surrogate, in µg/L.
C_{is} = Concentration of the internal standard, in µg/L.

10.5 Establishing the Calibration Function

Calibrations are modeled either as average response factors or as linear regression curves, using a systematic approach to select the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until calibration acceptance criteria are met. For dual column analysis the initial calibration criteria must be met on both columns.

10.5.1 Linear Calibration Using Average Response Factor

The response factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

NOTE: For samples requiring adherence to SW 846 Methods 8000B and 8000D (including South Carolina compliance work), forcing through zero is NOT allowed.

10.5.1.1 The average response factor is calculated as follows:

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$ Equation 2

Where:

RF_i = The response factor for the ith calibration level.
n = The number of calibration levels.

10.5.1.2 The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \text{Equation 3}$$

Where SD is the standard deviation of the average RF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}} \qquad \text{Equation 4}$$

10.5.2 Evaluation of the Average Response Factor

Plot the calibration curve using the average RF as the slope of a line that passes through the origin. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered.

Acceptance Criteria: The RSD must be ≤ 20%. SW-846 Method 8000B allows evaluation of the grand average across all compounds, but some programs (e.g., DoD/DOE QSM, Arizona and South Carolina require evaluation of each compound individually). Check project requirements.

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.5.3 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated is the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

Equation 5

$$y = ax + b$$

Where:

y = Instrument response (peak area or height).
x = Concentration of the target analyte in the calibration standard.
a = Slope of the line.
b = The y-intercept of the line.

For an internal standard calibration, the above equation takes the following form:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$ *Equation 6*

To calculate the concentration in an unknown sample extract, the regression equations 5 and 6 are solved for concentration, resulting in the following equations, where x and C_s are now the concentration of the target analyte in the unknown sample extract:

$$x = \frac{y - b}{a}$$ *Equation 7*

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$ *Equation 8*

10.5.4 Evaluation of the Linear Least-Squares Regression Calibration Function

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, the calibration should be repeated since a higher order regression is not allowed for this method.

The linear regression must have a correlation coefficient (r) ≥ 0.99. Some programs (e.g., DoD QSM 4.2) require a correlation coefficient ≥ 0.995. DoD/DOE QSM 5.0 and 5.1 require r^2 >0.99.

Corrective Action: If the correlation coefficient falls below the acceptance limit, the linear regression is unacceptable and the calibration should be repeated since a higher order regression is not allowed for this method.

10.5.5 Second-order regressions and polynomial regression fits of third order or higher are not allowed for this method.

10.5.6 Acceptance criteria independent of calibration model

10.10.1.1 Either of the procedures Percent Error or Relative Standard Error (RSE) may be used to determine calibration function acceptability for linear and non-linear curves. Both evaluate the difference between the measured and the true amounts or concentrations used to create the model.

$$\%\ Error = \frac{xi - xi}{xi} x\ 100$$

Where: $x'i$ = measured amount of the analyte at calibration level *i* in mass or concentration units and xi = the true amount of the analyte at calibration level *i* in mass or concentration units.
% Error should be ≤ 30%. For some data uses ≤ 50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error:

$$RSE = 100\,x \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i} \right]^2 \Big/ (n-p)}$$

Where: *Xi* = True amount of the calibration level I, in mass or concentration units
X'i = measured amount of analyte in calibration level I, in mass or concentration units
p = number of terms in the fitting equation (average = 1), linear – 2, quaratic – 3)
n = number of calibration points
RSE acceptance criteria for the calibration model is the same as the RSD limit for CF or RF in the determinative method. IF not defined in the method, use ± 20%.

10.6 Second-Source Initial Calibration Verification (ICV)

An ICV is prepared for each multipoint calibration. The stock standards are obtained from a source different than that of the standards used for the calibration. The preparation of the ICV standard is described in Section 7.5. The concentration of each Aroclor in the ICV is 0.25 µg/mL; the concentration of each surrogate is 0.125 µg/mL. The ICV standard is analyzed immediately following the completion of the initial calibration. If any changes are made to the calibration curve types then the ICV must be recalculated to the final form of the ICAL.

Acceptance Criteria: The result for the target analyte(s) in the ICV standard must be within ± 15% of the expected value. Method 8082A and DOD/DOE QSM 5.0 and 5.1 allow a control of ± 20%.

Corrective Action: If this is not achieved, the ICV standard, calibration standards, and instrument operating conditions should be checked. Correct any problems and rerun the ICV standard. If the ICV still fails to meet acceptance criteria, then repeat the ICAL.

10.7 Continuing Calibration Verification (CCV), 0.50 ug/mL, LVI 0.25 ug/mL.

10.7.1 12-Hour Calibration Verification

The 12-hour calibration verification sequence consists of, at a minimum, an instrument blank and the mid-level calibration standard. The 12-hour calibration verification sequence must be analyzed within 12 hours of the start of the initial calibration and at least once every 12 hours thereafter when samples are being analyzed. If more than 12 hours have elapsed since the injection of the last sample in the analytical sequence, a new analytical sequence must be started with a 12-hour calibration sequence.

NOTE: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.7.2 The mid-level calibration standard is analyzed as the continuing calibration verification (CCV) standard (see Section 7). At a minimum, this is analyzed after every 20 samples, including matrix spikes, LCSs, and method blanks. It may be appropriate to analyze a mid-level standard more frequently than every 12 hours. Some programs, specifically DoD/DOE QSM 5.0 and 5.1, require a CCV after every 10 field samples and at the end of the run. If 12 hours elapse, analyze the 12-hour standard sequence instead. Unless the program specifically requires a closing CCV (as above for DOD and DOE), a closing CCV is not required when using an internal standard. Check the projects requirements regarding the closing CCV.

If an aroclor other than 1016 or 1260 is identified in any sample then a CCV for the identified aroclor must be analyzed within 12 hours of the samples analysis. This CCV must meet proper CCV criteria.

For DOD/DOE QSM 5.0 or 5.1, the CCVs for aroclors other than 1016 and 1260 are only required before sample analysis.

10.7.3 RL Standard

It may be appropriate to analyze a standard prepared at or very near the reporting limit (RL) for the method between every 10 sample injections (see Section 7.7). This standard can be used to rule out false negatives in client samples in cases where the %D for one or more of the analytes in a bracketing CCV falls below the lower acceptance limit and the samples contain no analytes above the reporting limit. The results for the RL standard are not evaluated unless the previous CCV fails acceptance criteria or in the case of matrix effect to confirm the ability to see at the reporting limit.

NOTE: This procedure is not used when Method 8000C/D or DOD/DOE QSM 5.0 or 5.1 are required.

10.7.4 Acceptance Criteria for Continuing Calibration Verification (CCV)

10.7.4.1 Detected Analytes (≥ RL)

For any analyte detected at or above the reporting limit (RL) in client samples, the percent difference (%D) for that analyte in the preceding and following CCVs (i.e., bracketing CCVs) or 12-hour calibration, on the column used for quantitation, must be within ± 15%. Method 8082A and DOD/DOE QSM 5.0 and 5.1 require control of ± 20%. If a confirmation column is required (see Section 12.5), the CCV criteria must be met on both columns in order to confirm a detection. Both surrogates must pass in the CCV.

In some cases, the nature of the samples being analyzed may be the cause of a failing %D. When the %D for an analyte falls outside of acceptance criteria in the CCV, and that analyte is detected in any or all of the associated samples, then those samples must be reanalyzed to prove a matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect; if so desired by the client. Dilution will typically be required to meet DOD 4.2 or DoD/DOE QSM 5.0 or 5.1 criteria.

Refer to Section 12 for which result to report.

The %D is calculated as follows:

$$\%D = \frac{\text{Measured Conc} - \text{Theoretical Conc}}{\text{Theoretical Conc}} \times 100 \qquad \text{Equation 8}$$

10.7.4.2 Analytes Not Detected (< RL)

For any analyte not detected (ND) in client samples, the %D for that analyte in the bracketing CCVs should also be within acceptance criteria.

However, if the CCV %D exceeds the upper control of the acceptance criteria and the sample results are ND, it still may be possible to report sample results. In this case, the client should be consulted and an NCM written. Note that exceptions to an out of control CCV are not allowed for DoD/DOE QSM 5.0 or 5.1 unless prior approval is noted in the project instructions.

If the CCV %D falls below acceptance criteria and sample results are ND, but the target analytes are detected in the RL Standard, it may still be possible to report sample results, since the detection of the analyte(s) in the RL Standard indicate that there was sufficient sensitivity to detect the analyte(s) in the samples. In this case, the client should be consulted and an NCM written. This would only be used in cases where the matrix is affecting CCV recovery and dilution of the affected sample(s) is not an alternative.

DoD/DOE QSM 5.0 or 5.1 requires recalibration and reanalysis of all affected samples since the last acceptable CCV. As an alternative, the laboratory may analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, then the samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate: then reanalyze all affected samples since the last acceptable CCV.

If a DoD or DOE client accepts TestAmerica's Technical Specifications for DoD/DOE QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags required by the project.

10.8 Retention Time (RT) Windows

10.8.1 Determine the retention time (RT) windows for the 5 major peaks selected for each Aroclor (3 peaks for Aroclor 1221). The AR1016 windows will be used to establish retention time windows for AR1221, AR1016, AR1232, AR1242, and AR1248. The AR1260 windows will be used to establish retention time windows for AR1254, AR1260, AR1262, and AR1268.

10.8.2 Determine new RT windows each time a new column is installed or annually.

10.8.3 Evaluate the deviation from expected retention time for each analyte in at least three CCV and/or LCS samples spread over at least 72 hours.

10.8.4 If three days of analytical data are not available, use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCS in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

10.8.5 Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 min, in which case the window is set at 0.01 min. For example, if the maximum RT deviation for a specific analyte is 0.008 min, then the RT window is set at ± 0.012 min.

NOTE: For the Aroclors, the maximum deviation must be evaluated for each of the 3-5 major peaks used for sample calculations.

10.8.6 Retention time windows for analytes of interest must not overlap.

10.9 Ongoing Evaluation of Retention Time Windows

10.9.1 Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

10.9.1.1 Matrix spike analytes may fall outside the retention time window if there is a large non-target pak coeluting with the analyte in the matrix spike.

10.9.2 If any analytes fall outside the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

10.9.3 Retention time windows should be reliably narrower than ± 0.03 min. If RT windows wider than this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained. Method 8000B requires a new retention time window study be performed annually or when the analytical column from a new vendor or different stationary phase is used. 8000C and 8000D also require a new study when the column is clipped during maintenance.

10.9.4 Sample Retention Time Criteria

The surrogate must fall within the established RT window. Target analyte peaks must be within the established RT window to be reported as such. If the surrogate RT indicates a RT shift, it may be possible to accept a target analyte peak if it has shifted in the same direction as the surrogate peak. The presence of a definitive aroclor pattern will be positive evidence

of a hit and may supersede RT window criteria. An NCM should be written to explain this case.

10.9.5 Daily Retention Time Windows

The centers of the retention time windows are adjusted at the beginning of each analytical sequence based on the daily initial CCV or the RT marker mix.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

11.3 Sample Preparation

11.3.1 Sample preparation for aqueous samples is described in SOP DV-OP-0006.

11.3.2 Sample preparation for solid samples is described in SOPs DV-OP-0016 and DV-OP-0015.

11.3.3 Cleanup and concentration of sample extracts are described in SOP DV-OP-0007. Note that it is highly recommended that all samples be checked for sulfur and cleaned up if necessary before the samples are analyzed on the instrument. Sulfur can contaminate the column and hinder the quantification of certain compounds.

11.3.4 For water samples, a 1 L extraction is brought to a final extract volume in hexane of 10 mL. For water samples extracted from a 250 mL volume and analyzed by the LVI method the final volume is 5 mL.

11.3.5 For solid samples, a 30 g sample is brought to a final volume of 10 mL of hexane.

11.3.6 Wipe samples are processed as a modified soil and the final volume of these extracts is 10 mL of hexane.

11.3.7 Oil samples are processed as a waste. Typically 1 g of oil is brought to 10 mL with hexane.

11.3.8 Use hexane to dilute sample extracts, if necessary.

11.4 Instrument Troubleshooting and Maintenance

Before the start of any daily sequence the instrument system should be evaluated for possible maintenance.

11.4.1 If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control.

11.4.2 The injector septum should be changed after about 200 injections have been completed.

11.4.3 If the last CCV that was analyzed indicated a high response then a simple liner change is typically sufficient to bring the system back into control.

11.4.4 Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column.

11.4.5 A reduced response may indicate that the system needs to be evaluated for leaks.

11.4.6 Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated. The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed.

11.4.7 Re-calibration should not be used to correct for maintenance related issues. Always document any maintenance procedure in the maintenance logbook.

11.5 Gas Chromatography

Chromatographic conditions for this method are presented in Tables 2 and LVI-2. Use the ChemStation interface to establish instrument operating conditions for the GC. Raw data obtained by the ChemStation software is transferred to the Chrom DB database for further processing. The data analysis method, including peak processing and integration parameters, calibration, RT windows, and compound identification parameters, is set up in the Chrom DB software.

11.6 Sample Introduction

All extracts and standards are allowed to warm to room temperature before injection. An autosampler is used to introduce samples into the chromatographic system by direct injection of 1 or 2 µL of the sample extract. For LVI analysis 2 µL of sample extract is introduced into the chromatographic system. Samples, standards, and QC samples must be introduced using the same procedure. Use the Chrom worklist to set up and run the analytical sequence. Sample injection and analysis are automated and may proceed unattended.

11.7 Analytical Sequence

An analytical sequence starts with a minimum five-level initial calibration (ICAL) or a daily calibration verification. Refer to Tables 3 and LVI-3 for the calibration levels

used.

11.7.1 The daily calibration verification includes analysis of the 12-hour calibration sequence (Section 10.7.1) and updating the retention time windows (Section 10.8.7)

11.7.2 If there is a break in the analytical sequence of greater than 12 hours, a new analytical sequence must be started with a daily calibration verification. Any samples that were not bracketed by a closing CCV (when required) must be reanalyzed in the new 12 hour sequence.

11.7.3 The following is a typical analytical sequence for routine sample analysis:

- Primer (Injection of any standard that contains any of the analytes to establish the stability of the chromatographic system.)
- RT marker mix.
- Hexane instrument blank.
- Daily initial CCV (Unless an ICAL is performed, which is immediately followed by the second-source initial calibration verification.)
- 10 sample injections (The first set of samples analyzed usually includes the method blank and the LCS, and may include matrix spikes.)
- CCV
- Followed by cycles of 10 sample injections and a CCV, as needed
- Closing CCV, instrument blank, and RL Standard

11.8 Retention Times

The centers of the RT windows determined in section 10.8 are adjusted to the RT of each individual peak as determined in the 12-hour calibration verification. The RT window must be updated at the beginning of each analytical sequence.

11.9 When a sample result exceeds the upper calibration range, then that sample extract is diluted to obtain a result in the upper half of the calibration range and reanalyzed. Any samples that were analyzed immediately following the high sample are evaluated for carryover. Typically carryover is not observed until the sample concentration is well above the upper calibration level. If the samples had target analyte detections at or above the RL, the samples must be reanalyzed to rule out carryover.

11.10 Upon completion of the analytical sequence, transfer the raw chromatography data to the Chrom database for further processing. Review chromatograms online and determine whether manual data manipulations are necessary. All manual integrations must be justified and documented. See DV-QA-011P for requirements for manual integration. Manual integrations may be processed using an automated macro, which prints the before and after chromatograms and the reason for the change, and attaches the analyst's electronic signature. Alternatively, the manual integration may be processed manually. In the latter case, print both the both the

before and after chromatograms and record the reason for the change and initial and date the after chromatogram. Before and after chromatograms must be of sufficient scale to allow an independent reviewer to evaluate the manual integration. These chromatograms must be scanned into the documents section of the LIMS system.

11.11 Compile the raw data for all the samples and QC samples in a batch. The analytical batch is defined as containing no more than 20 samples, which include field samples and the MS and MSD.

11.11.1 The data package should consist of the checklist, sequence(s), ICAL cover, ICAL summary and history used for data quantitation and the prep batch paperwork.

11.11.2 Perform a level 1 data review and document the review on the data review checklist, GC Data Review Checklist/Batch Summary (See SOP DV-QA-0020.)

11.11.3 Submit the data package and review checklist to the Data Review Group for the level 2 review. All manual integrations must be evaluated by the peer reviewer and this review must be documented by date and initial on the level 2 review checklist. The level 2 review is documented on the review checklist initiated at the level 1 review. The data review process is explained in SOP DV-QA-0020.

12.0 Calculations / Data Reduction

12.1 Detailed equations can be found in the Corporate SOP CA-Q-P-003 *Calibration Curves and the Selection of Calibration Points* and under the public folder, *Arizona Calibration Training*.

12.2 Qualitative Identification of Aroclors

Retention time windows are used for identification of Aroclors, but the "fingerprint" produced by major peaks of those analytes in the standard is used in tandem with the retention times for identification. The ratios of the areas of the major peaks are also taken into consideration. Identification may be made even if the retention times of the peaks in the sample fall outside of the retention time windows of the standard, if in the analyst's judgment the fingerprint (retention time and peak ratios) resembles the standard chromatogram. A NCM must be written to explain the deviation from the expected RT criteria. Project specific allowance for DoD/DOE QSM 5.0 or 5.1 is required for the TestAmerica approach to the RT shift for aroclors such that the retention time shift occurs at a later point in the sequence. Samples with detections must be reanalyzed after the analysis of a retention time marker for any aroclors that are detected and show a RT shift unless project specific requirements allow for reporting data with a NCM.

12.3 Quantitation of Aroclors

Quantitation of Aroclors is accomplished using 5 major peaks (3 peaks for Aroclor 1221). If there is an interference that affects the accuracy of results, the analyst

may use as few as 3 major peaks (2 peaks for Aroclor 1221). The same peaks that are used for sample quantitation must be used for standards and QC quantitation.

12.4 Second column confirmation of Aroclors is performed only when requested by the client, because the appearance of the multiple peaks in the sample usually serves as a confirmation of analyte presence.

NOTE: DoD/DOE QSM 5.0 and 5.1 projects require the use of second-column confirmation of Aroclors unless the project work plans (SOW, SAP, QAPP, etc.) specify single-column analysis.

NOTE: South Carolina requires second column confirmation.

NOTE: Method 8082A indicates that second column confirmation is necessary when the sample composition is not well characterized.

12.5 Dual Column Quantitation

NOTE: This section is included for those clients/projects that require dual column confirmation.

12.5.1 A primary column is designated when dual column analysis is performed. If the continuing calibration fails for one of the columns then the appropriate corrective action must be taken. The result from the primary column is normally reported. The result from the secondary (confirmatory) column is reported if any of the following is true:

12.5.1.1 There is obvious chromatographic interference on the primary column.

12.5.1.2 The difference between the result for the primary column and the result for the secondary column is > 40% and chromatographic interference is evident on the primary column.

12.5.1.3 For DoD QSM 4.2 or DOD/DOE QSM 5.0 work, calibration and QC criteria for the second column are the same as for the initial or primary column analysis.

12.5.2 Dual Column Results With > 40% RPD

12.5.2.1 If the relative percent difference (RPD) between the responses on the two columns is greater than 40%, the higher of the two results is reported unless there is obvious interference documented on the chromatogram. Method 8000C/D requires that the lower result be reported. For DOD/DOE QSM 5.0 or 5.1, use project-specific or method requirements to determine reporting convention. If none specified, report from the primary column.

12.5.2.2 If there is visible positive interference, e.g., co-eluting peaks, elevated baseline, etc., for one column and not the other, then

report the results from the column without the interference with the appropriate data qualifier flag, footnote, and/or narrative comment in the final report.

12.5.2.3 If there is visible positive interference for both columns, then report the lower of the two results with the appropriate flag, footnote, and/or narrative comment in the final report.

12.5.2.4 The RPD between two results is calculated using the following equation:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 14}$$

Where R_1 is the result for the first column and R_2 is the result for the second column.

12.5.3 Reporting Total Aroclors for dual column reporting or from multiple runs.If dual column quantitation is requested or if the results from the individual Aroclors are in multiple runs, then total aroclors will be calculated from the primary results only using the TALS method code Total_PCB.

12.6 Surrogate Recovery

12.6.1 Surrogate recovery results are calculated and reported for decachlorobiphenyl (DCB).

12.6.2 In cases where the addition of the surrogate tetrachloro-*m*-xylene (TCMX) is required, its recovery is calculated and reported. In cases where both surrogates are added and recoveries calculated, the recovery of each surrogate is evaluated and corrective action must be taken if either surrogate recovers outside of the established control limits and matrix interference is not evident. Depending on project requirements, corrective action may be necessary only if DCB and TCMX are both outside of acceptance limits.

12.7 Calibration Range and Sample Dilutions

12.7.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for any analyte, they must be reanalyzed to rule out carryover unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when blanks or other samples were analyzed after a sample with similar high concentration or when the detection in the sample with suspected carryover is much higher than the expected amount of the

carryover (i.e., the suspect sample's concentration is similar to or higher than the sample run previous to it). It may also be necessary to dilute samples because of matrix interferences.

12.7.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

12.7.3 Guidance for Dilutions Due to Matrix Interference

If the sample is initially run at a dilution and only minor matrix peaks are present, then the sample should be reanalyzed at a more concentrated dilution. Analyst judgment is required to determine the most concentrated dilution that will not result in instrument contamination. Ideally, the dilution chosen will make the response of the matrix interferences equal to approximately half the response of the mid-level calibration standard.

12.7.4 Reporting Dilutions

Some programs (e.g., South Carolina, DoD and DOE) and some projects require reporting of multiple dilutions (check Method Comments in LIMS). In other cases, the most concentrated dilution with no target compounds above the calibration range will be reported.

12.8 Interferences are Observed in Samples

12.8.1 Dual column analysis does not entirely eliminate interfering compounds. Complex samples with high background levels of interfering organic compounds can produce false positive and/or false negative results. The analyst must use appropriate judgment to take action as the situation warrants.

12.8.2 Suspected Negative Interferences

If peak detection is prevented by interferences, further cleanup should be attempted. Elevation of reporting levels and/or lack of positive identification must be addressed in the case narrative.

12.8.3 Suspected Positive Interferences

If no further cleanup is reasonable and interferences are evident that are suspected of causing false positive results, consult with the laboratory Project Manager to determine if analysis using additional confirmation techniques is appropriate for the project. Use of additional confirmation columns is another possible option. At a minimum, the Data Review Template prepared by the analyst should include the following comment for inclusion in the case narrative:

"Based on review of the chromatograms for samples _____, it is my opinion that the evident interferences may be causing false results.

Date _____ Analyst _____"

12.9 Identifying and Reporting PCBs

12.9.1 In samples where the PCB pattern matches an individual Aroclor reasonably well, the samples should be quantified and reported as usual. When there are numerous PCB peaks present but there are no good matches to any individual Aroclor, choose the Aroclor (or Aroclors) that most closely match the sample and quantify the peaks as that Aroclor. The sample should not be reported as "not detect" based solely on the absence of a good match to a single Aroclor mixture. Multiple Aroclors should only be reported if their patterns are reasonably well separated. For example, 1232 and 1254 could be reported together, but not 1242 and 1248. See Attachment 1 for additional information on identifying Aroclors.

NOTE: When reporting and quantifying PCBs that do not closely match an Aroclor standard, it is absolutely essential and mandatory that this is explained in the report narrative. The analyst must write a NCM to explain their judgment for reporting PCBs in these instances.

12.9.2 Some example text that can be used in the report narrative is presented below:

Sample XXXX appears to contain PCBs based on the presence of numerous PCB peaks. However, due to weathering or other environmental processes, the PCBs in the sample do not closely match any of the Aroclor standards we use to calibrate our instruments. We quantified and reported the sample as Aroclor ZZZZ (or as a mixture of Aroclors ZZZZ and YYYY). Due to the poor match with the Aroclor standard(s), there is increased qualitative and quantitative uncertainty associated with this result. This approach is consistent with the guidance in section 7.9.3 of SW846 method 8082A. If these results do not meet the needs of your project then we would suggest a further analysis of the sample. Depending on the objectives, this may include congener-specific analysis by 8082A; or analysis by a more specific method (e.g., method 1668 or an adaptation of method 8270) for PCB congeners or PCB homolog totals.

12.9.3 Sample Matrix Issues

12.9.3.1 In some cases when analyzing for multi-component analytes, the sample matrix is so complex that it would obliterate any possible pattern that would allow us to identify the analyte. When this happens, it is true that the analyte is not detected at the normal detection limit. However, it is true that we could not have detected the analyte at the normal detection limit. Even if the analyte was present, we would not be able to recognize it.

12.9.3.1 When this occurs, the sample must be analyzed at a dilution that would allow us to detect the analyte, and the reporting limit should be the one appropriate for that dilution. Reporting a non-detect at the normal reporting limit is not an acceptable practice.

12.9.3.2 Some clients may insist on ND reporting if the patterns are not clear. In that event, add information to the project file to indicate that the information in this guidance have been communicated to the client, together with the client's instructions. In addition, in the event of a poor match to patterns, a narrative comment as suggested in the previous paragraph is still required.

12.9.4 Background on PCBs

12.9.4.1 PCBs were widely used in a variety of products prior to being banned in the 1970's. The most common usages were in electric motors and transformers. They were manufactured by gas phase chlorination of a biphenyl molecule. The nomenclature, in general, describes the weight percent of chlorine in the final product. Thus, Aroclor 1254 was produced by chlorinating a quantity of biphenyl until the resulting product was 54% chlorine by weight. Aroclor 1242 was 42% chlorine by weight.

12.9.4.2 PCBs were manufactured in batch processes, so there were slight variations between batches, but in general each Aroclor had a very reproducible pattern of chlorinated biphenyl isomers (congeners). With few exceptions, when we detect PCBs in the environment the initial contaminant was one of the Aroclors.

12.9.4.3 The one exception to the nomenclature of the Aroclors is Aroclor 1016. In the 1960's researchers started to find PCBs in fish tissue in the Great Lakes. The primary congeners appearing in the fish were pentachlorobiphenyls. The manufactures of PCBs devised a synthetic process that created an Aroclor with very similar properties to Aroclor 1242, but minimized the formation of pentachlorobiphenyl molecule. Aroclor 1016 41% chlorine by weight and as result it can be difficult to distinguish from 1242.

12.9.4.4 While the pattern of congeners was quite reproducible in the pure products once in the environment the pattern changes. The lesser chlorinated PCBs are more water soluble and are more volatile, while the more highly chlorinated PCBs bind to solids and sediments more strongly. As examples, landfill gas condensates tend to have a bias toward the lesser chlorinated congeners because they are more volatile. River sediments near source of PCBs tend to have a bias toward the more highly chlorinated congeners because the accompanying lesser chlorinated congeners were more water soluble. Downstream from the source of contamination, however, there will be a bias toward the less chlorinated congeners because the more heavily chlorinated congeners were trapped in the sediments near the

outfall. Anaerobic and aerobic microbial degradation reduce the concentrations of some congeners and an increase in concentrations of others. Although they are rarely the primary mechanisms, oxidative and photolytic processes are also selective, impacting some congeners more than others.

12.9.4.5 As a result, PCBs in the environment rarely have an exact match to the Aroclor standards that we use to calibrate our instruments. There is inevitably some level of judgment required to choose the Aroclor that has the best match to the sample in questions. Sometimes this is straightforward, but other times the judgment is difficult and can be controversial. In the worst cases, we can have situations where there are clearly PCB peaks throughout a chromatogram, but there is no good match with any of the Aroclors. It is recommended that at a minimum there must be some peak groupings present that are characteristic of an aroclor pattern in order to indicate a positive detection.

12.10 Calculations

12.10.1 Concentration of Analyte in Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.5 for details on establishing the calibration function):

12.10.1.1 Average Response Factor:

$$C_e = \frac{A_e}{RF} \qquad \text{Equation 12}$$

12.10.1.2 Linear Regression:

$$C_e = \frac{[A_e - b]}{a} \qquad \text{Equation 13}$$

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_e = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

12.10.2 Concentration of Analyte in Original Sample

The concentration of the analyte in the original sample is calculated as follows:

$$C_{sample} = \frac{C_e}{1000\ \frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF$$ Equation 14

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).

C_e = Concentration of analyte in sample extract injected in GC (ng/mL).

$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.

V_e = Volume of sample extract (mL).

V_s = Volume (or weight) of original sample (L or kg).

DF = Dilution Factor (post extraction dilutions)

12.10.3 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\%$$ *Equation 15*

12.10.4 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD } \%\text{Recovery} = \left(\frac{SSR - SR}{SA}\right) \times 100\%$$ *Equation 16*

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.10.5 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\%$$ *Equation 17*

Where *R1* is the result for the MS and *R2* is the result for the MSD.

12.11 All data are subject to two levels of review, which is documented on a checklist, as described in SOP DV-QA-0020.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

An initial method detection limit study is performed for each analyte and each sample matrix type in accordance with Policy CA-Q-S-006. Each of the other aroclors have an MDLV performed annually to verify the MDL. For DoD, DOE and Texas TRRP projects, AR_1660 MDLVs and LOQVs are performed quarterly. MDLs and LOQs are stored in LIMS.

13.2 MDLV and LOQV

13.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes at the programmatically required spike amounts.

13.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

13.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test

need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention.

14.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Waste hexane solvent: Flammable Solvent – Waste Stream C

15.2.2 Vials containing extracts in hexane: Expired Extract Vials – Waste Stream A

15.2.3 Concentrated sulfuric acid and hexane from sample cleanup: Concentrated Acids with Organics - Waste Stream V

15.2.4 Expired reagents and standards – Contact Waste Coordinator

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.2.5 Samples containing polychlorinated biphenyls (PCB's) at concentrations ≥50 ppm are regulated under the Toxic Substance Control Act (TSCA) and must be segregated from all other waste streams. Analysts are responsible for contacting the Group Leader, Sample Control, and the Waste Coordinator immediately if a sample falls into the TSCA category.

16.0 References / Cross-References

16.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.2 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.1.3 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.1.4 Method 3546, Microwave Extraction, Revision 0, February 2006.

16.1.5 Method 3580A, Waste Dilution, Revision 1, July 1992.

16.1.6 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.1.7 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.1.8 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 0, December, 1996.

16.1.9 Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Revision 1, February 2007

16.1.10 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.1.11 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.12 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

16.1.13 Method 8000D, Determinative Chromatographic Separations, Revision 5, March 2018.

16.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/25/2010.

16.3 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Final Version 5.0, July 2013.

16.4 Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.1, July 2017

17.0 Method Modifications

Item	Method	Modification
1	8000B	Method 8000 allows for use of a second order or third order calibration curve. TestAmerica Denver does not allow for any curvilinear calibrations for the analysis of Aroclors.
2	8000C/D	Method 8000C and D require that new retention time windows be established if a GC column has been shortened during maintenance. Given the matrices of the samples the laboratory receives, and the number of times the GC column may require clipping, TestAmerica Denver does not perform an RT study after clipping a column. RT studies done by the laboratory show that, historically, RT windows have not been greater than the method allowed 0.03 minutes. The lab defaults to a 0.03 minute RT window as allowed by the method.

18.0 Attachments

Table 1: Analyte List and Standard Reporting Limits
Table 2: Typical Instrument Conditions
Table 3: Calibration Levels (µg/mL)
Table 1-LVI: Analyte List and Standard Reporting Limits using Large Volume Injection
Table 2-LVI: Typical Instrument Conditions using Large Volume Injection
Table 3-LVI: Calibration Levels (µg/mL) using Large Volume Injection
Attachment 1: Aroclor Identification 101
Attachment 2: Example of Minimum Resolution Requirement for Aroclor 1260 Triplet

19.0 Revision History

Revision 16, March 03, 2020

- Annual Review
- Updated copyright information

Revision 15, January 4, 2019

- Added sections 13.1 and 3.8 in regards to LLOQ checks and standards.

Revision 14, October 31, 2018

- Added references to SW 846 Method 8000D

- Added explanations and calculations for % Error and Relative Standard Error
- Added method modification applicable to RT study requirements after column clipping
- Added references to South Carolina requirements
- Correct last sentence in the copyright information
- Updated the MDL SP number

Revision 13, July 31, 2018

- Annual Review

Revision 12, June 30, 2017

- Annual Review
- Updated references to DOD/DOE QSM 5.1 throughout in addition to existing QSM 5.0.
- Revised section 9.3 to reflect current requirements under DOD/DOE QSM that if no MS/MSD available, LCSD is performed in addition to LCS.
- Revised section 10.7.2 to reference both 1016 and 1260 rather than the combined nomenclature used internally by the laboratory.
- Updated Section 11.11.13 to reflect current practice for documenting review of manual integrations.
- Added references to section 16 for Method 8000D and DOD/DOE QSM 5.1.

Revision 11, June 3, 2016

- Annual Review
- Adjusted source references and concentrations in Section 7 as required to reflect current practice
- Updated Section 9.1 to reflect current practice
- Updated Section 9.6 to reflect current policy for over-range MS/MSD.
- Added "midpoint" to IS acceptance criteria in Section 9.8 to clarify standard used for evaluation of IS and updated acceptance window for IS recovery.
- Corrected resolution requirement criteria in Section 10.3.2.1
- Updated reference to corporate SOP on calibration curves in Section 10.3.1 and 12.1. Document was revised and number was changed.
- Added sentence to section 10.3.5 to reflect that all quantitations of identified aroclors are performed using a calibration curve (minimum five points).
- Added clarification to Section 10.3.8 for minimum height of peaks selected for quantitation relative to the largest peak in the aroclor (except 1268 at 10%)
- Added clarifications to paragraphs in Section 10.7.4.1
- Revised Section 10.8 and 10.9 to comply with Policy CA-T-P-005 for determining RT windows. This is intended to assist in minimizing false positive results.
- Revised language in Section 12.2 to explain when analyst interpretation of PCBs with a RT shift can be used
- Revised Section 13 to reflect current practice
- Replaced calibration factor with response factor throughout as this is now an internal standard method
- Editorial and formatting changes throughout

Revision 10, April 30, 2015

- Annual Review

- Added Internal Standard quantitation method throughout including
  - Section 7.3.1 – added description of internal standard stock
  - Section 7.11: IS solution,
  - Section 9.8: IS Acceptance criteria and corrective action
  - Section 10.3.2: Note regarding resolution check for Aroclor 1260
  - Section 10.4: Internal Standard Calibration
  - Incorporated equations for IS throughout Section 10.5
  - Added to section 10.7.2 that closing CCV may not be required with use of IS
  - Added requirement to run CCV for any identified aroclor if other than 1660
  - Section 10.8.7: Added use of RT marker for all representative aroclor peaks
- Corrected statement in Section 10.8.9 regarding shift noted by surrogate
- Added in Section 10.8.10 that RT windows can be set by RT marker
- Made additions to Section 11.7 for Analytical Sequence based on IS method
- Removed original Item 1 in Method Modifications table; renumbered.
- Added new Attachment 2

Revision 9, June 30, 2014

- Annual Review
- Updated the following sections to add include more detail and/or reflect correct practices. Sections: 1.2, 1.3, 2.1.2, 4.1, 6.1, 7.3.1, 7.4.1 (table), 7.4.2 (table), 7.5.1, 7.8, 9.1.2, 9.3, 9.5 (corrective action), 9.6 (corrective action), 10.1.2, 10.1.3, 10.3.6.4, 10.4, 10.5.4 (acceptance criteria), 10.6, 10.7.2, 10.7.4.1, 10.7.4.2, 11.5, 11.6, 11.9, 11.10.3, 12.2 and 12.3
- Corrected formatting and grammatical errors
- Added section 11.4 – Troubleshooting
- Additional references added to section 16
- Added total Aroclor calculation when summing individual Aroclors from dual column or different runs to section 12.5.3

Revision 8, October 30, 2013

- Updated sections 7.3,and 7.5 for the use of standard/standards
- Updated section 7.4.2 to correct table for CCV preparation
- Updated section 10.7 to add LVI CCV concentration
- Updated Table 2 and 2-LVI for injection volumes
- Added LVI tables to section 7.4

Revision 7, June 30, 2013

- Added section 2.1.2
- Added statement to Section 2.2 regarding use of 2nd column confirmation for DoD and method 80082A while presences of multiple peaks characteristic of an aroclor otherwise serve as confirmation when 2nd column is not required.
- Added sentence to Section 5.1.4 regarding procedures for handling radioactive materials
- Updated columns specified in Section 6.3
- Added injection port liners to section 6.4.2
- Added requirements for sample containers and sample volume for LVI to section 8.0
- Added clarifications to Corrective Action Section in Section 9.7 for evaluation and reporting of samples based on surrogate recoveries.

- Replaced references to Target software with Chrom software to reflect current practice
- Added dilution solvent (hexane) and extract final volumes for each matrix analyzed to section 10.3
- Removed note regarding restriction of use of Grand Mean as Method 8082 does not use the grand mean approach to CCV acceptance
- Added requirements for initial and final volume for LVI (Section 11.3.4)
- Added requirements for LVI injection volume (4 µL) to Section 11.5
- Added requirement for reporting lower result by Method 8000C (Section 12.5.2.1)
- Removed statement in section 12.9.2 duplicated in section 12.9.3.3
- Revised Table 2 to reflect current practice
- Revised Tables 1-LVI, 2-LVI, and 3-LVI to reflect current practice

Revision 6, June 15, 2012

- Added Tables 1-LVI, 2-LVI, and 3-LVI for large volume injection

Revision 5.1, January 16, 2012

- Changed extraction holding time for water and solid to 1 year with exclusion for California, Connecticut, Pennsylvania and South Carolina (Section 8).
- Reformatted paragraphs throughout

Revision 5, December 2011

- Combined SOP DV-GC-0021 and DV-GC-0030 Rev. 0.2. Upon implementation of this revision of SOP DV-GC-0021, SOP DV-GC-0030 will be deactivated.
- Added details for analysis of polychlorinated terphenyls by this procedure (sections 1, 3, 7 and Table 1).
- Updated Section 6 to include reference to master list of documents, software and hardware and volumetric flasks.
- Updated refrigerator temperature references from 4 $\pm$ 2°C to 0-6°C throughout.
- Updated vendors and catalog numbers for standards (Section 7)
- Updated Section 9 for consistency with SOP DV-QA-003P.
- Added calibration section to describe calibration models.
- Revised Procedure (new section 11) to be consistent with other SOPs revised in the last year.
- Added detail about review process (Section 11.8)
- Revised Calculations section (new section 12) to address dual column quantitation, sample dilution, and recovery calculations.
- Revised section numbers for previous sections 12-18.
- Updated Method Modifications section
- Revised Table 1 and Table 2

Earlier revision histories have been archived and are available upon request.

Table 1. Analyte List and Standard Reporting Limits

Compound	Water Reporting Limit (µg/L)	Soil Reporting Limit (µg/kg)
Aroclor 1016	1.0	33
Aroclor 1221	1.0	47
Aroclor 1232	1.0	33
Aroclor 1242	1.0	33
Aroclor 1248	1.0	33
Aroclor 1254	1.0	33
Aroclor 1260	1.0	33
Aroclor 1262	1.0	33
Aroclor 1268	1.0	33
PCT 5432	0.5	50
PCT 5442	0.5	75
PCT 5460	0.5	50

Table 2. Typical Instrument Conditions

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument W 125 °C for 1 minute 25 °C/min to 175 °C 10 °C/min to 275 °C 30 °C/min to 320 °C for 5 minutes Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	CLPI, 30 m x 0.32 mm id, 0.5 µm
Column 2:	CLPII, 30 m x 0.32 mm id, 0.25 µm
Injection:	2 µL 8082, 4 µL 8082 LVI
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek or J&W or Supelco glass tee, single gooseneck liner

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Table 3. Calibration Levels (µg/mL)

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1221	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1232	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1242	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1248	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1254	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1260	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1262	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Aroclor 1268	0.025	0.05	0.1	0.25	0.5	0.75	1.0
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05
Decachlorobiphenyl	0.00125	0.0025	0.005	0.0125	0.025	0.0375	0.05

Table 1-LVI. Analyte List and Standard Reporting Limits for Large Volume Injection

Compound	Water Reporting Limit (µg/L)
Aroclor 1016	1.0
Aroclor 1221	1.0
Aroclor 1232	1.0
Aroclor 1242	1.0
Aroclor 1248	1.0
Aroclor 1254	1.0
Aroclor 1260	1.0
Aroclor 1262	1.0
Aroclor 1268	1.0
PCT 5432	NA
PCT 5442	NA
PCT 5460	NA

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Table 2-LVI. Typical Instrument Conditions for Large Volume Injection

Parameter	Recommended Conditions
Injection Port Temperature:	250 °C
Detector Temperature:	325 °C
Temperature Program:	Instrument P3 125 °C for 1.25 minutes 30 °C/min to 180 °C 12 °C/min to 280 °C 15 °C/min to 320 °C for 2.6 minutes
Column 1:	Restek Rtx-CLPesticides 30m X 0.32 mm id, 0.5 µm (Cat# 11139 or equivalent)
Column 2:	Restek Rtx-CLPesticides2 30m X 0.32 mm id, 0.5 µm(Cat# 11324 or equivalent)
Injection Volume:	1 µL 8082, 2 µL 8082 LVI
Carrier Gas:	Hydrogen
Make-up Gas:	Nitrogen
Y-splitter:	Restek Universal Presstight Connector (Cat# 20400 or equivalent)
Injection Port Liner:	Agilent 5190-2293 90011 or equivalent

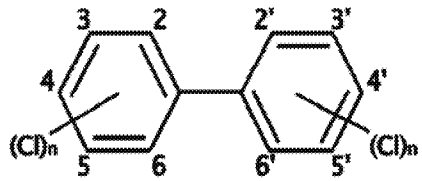
Table 3-LVI. Calibration Levels (µg/mL) for Large Volume Injection

Aroclors	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Aroclor 1016	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1221	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1232	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1242	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1248	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1254	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1260	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1262	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Aroclor 1268	0.0125	0.025	0.050	0.125	0.250	0.375	0.50
Surrogates are included in the AR_1660 calibration mix at the following levels:							
Tetrachloro-m-xylene	0.00063	0.00125	0.0025	0.00625	0.0125	0.0188	0.025
Decachlorobiphenyl	0.00063	0.00125	0.0025	0.00625	0.0125	0.0188	0.025

Attachment 1.

Aroclor identification 101

It can be difficult to correctly identify which Aroclor is present in a sample. This document provides a few guidelines. We are calling this document Aroclor identification 101 not because it is simple, but because Aroclor identification 201 and 301 (mixed, weathered Aroclors) are much more difficult still (sort of like P-Chem!)



First, we should consider what Aroclors actually are: They are mixtures of polychlorinated biphenyls.

Each phenyl ring can accommodate between zero and 5 chlorines. There are 209 possible isomers with 1-10 chlorines (the surrogate decachlorobiphenyl is the fully chlorinated molecule). Of these, about 130 are present in various Aroclor mixes, accounting for the complexity of the chromatograms. The first two digits of the Aroclor number refers to the number of carbon atoms, the last two refer to the degree of chlorination. Thus Aroclor 1248 has 12 carbon atoms in each molecule, and 48% chlorine by mass. So, as the last two digits increase, the overall degree of chlorination increases, the volatility decreases, and the pattern of peaks moves later in the chromatogram. Arochlor 1248 consists of approximately 1% monochlorobiphenyl, 13% dichlorobiphenyl, 45% trichlorobiphenyl, 31% tetrachlorobiphenyl and 10% pentachlorobiphenyl.

It is estimated that 1.25 billion pounds of PCBs were produced until Monsanto ceased production in 1977. PCBs are very persistent, so much of this material is still present in the environment.

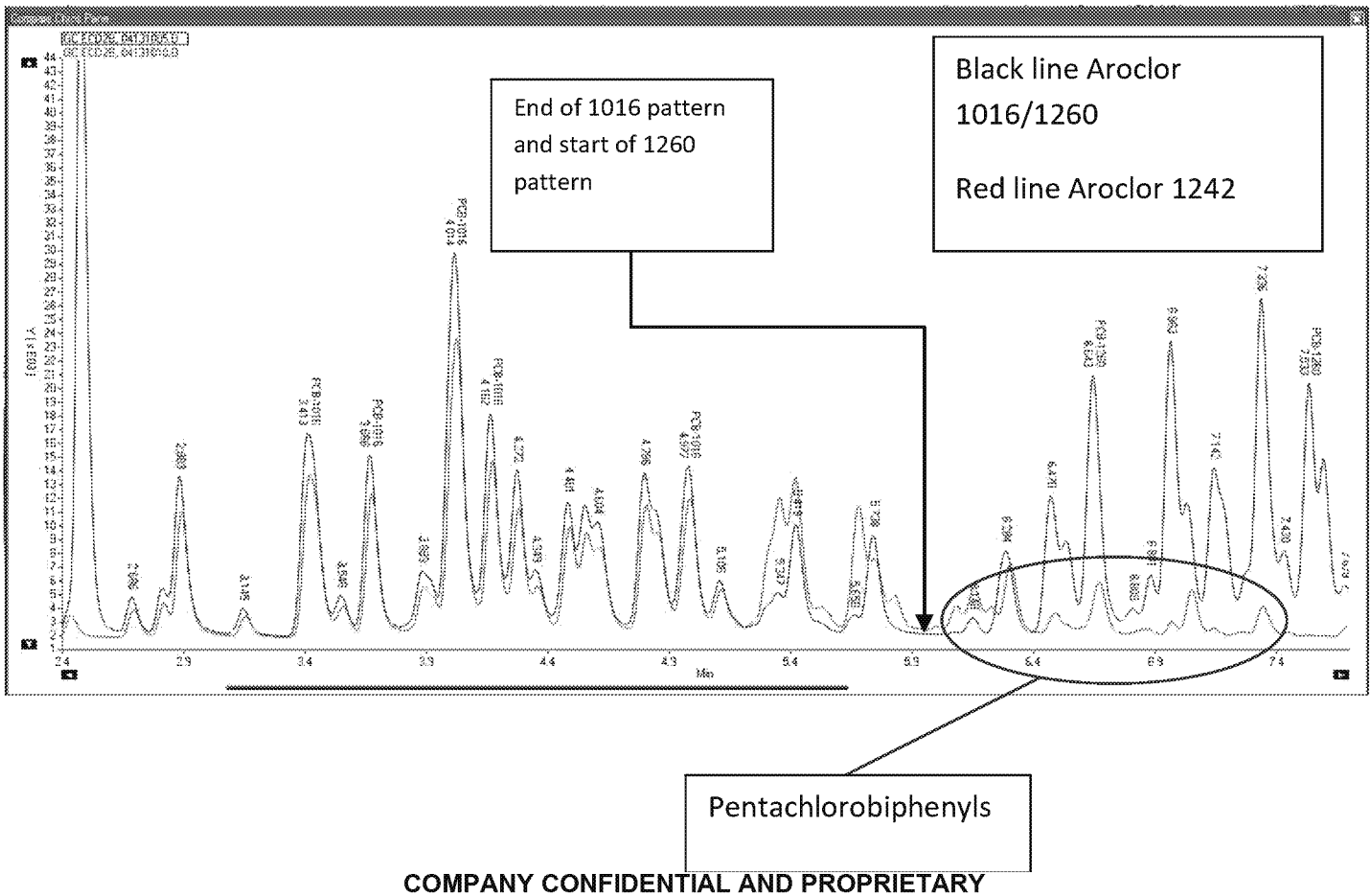
Example Chromatograms

In the following examples I'll refer to retention times frequently – your retention times will of course be different because of different chromatographic conditions but the same principles apply.

Attachment 1. Aroclor Identification 101 (Continued)

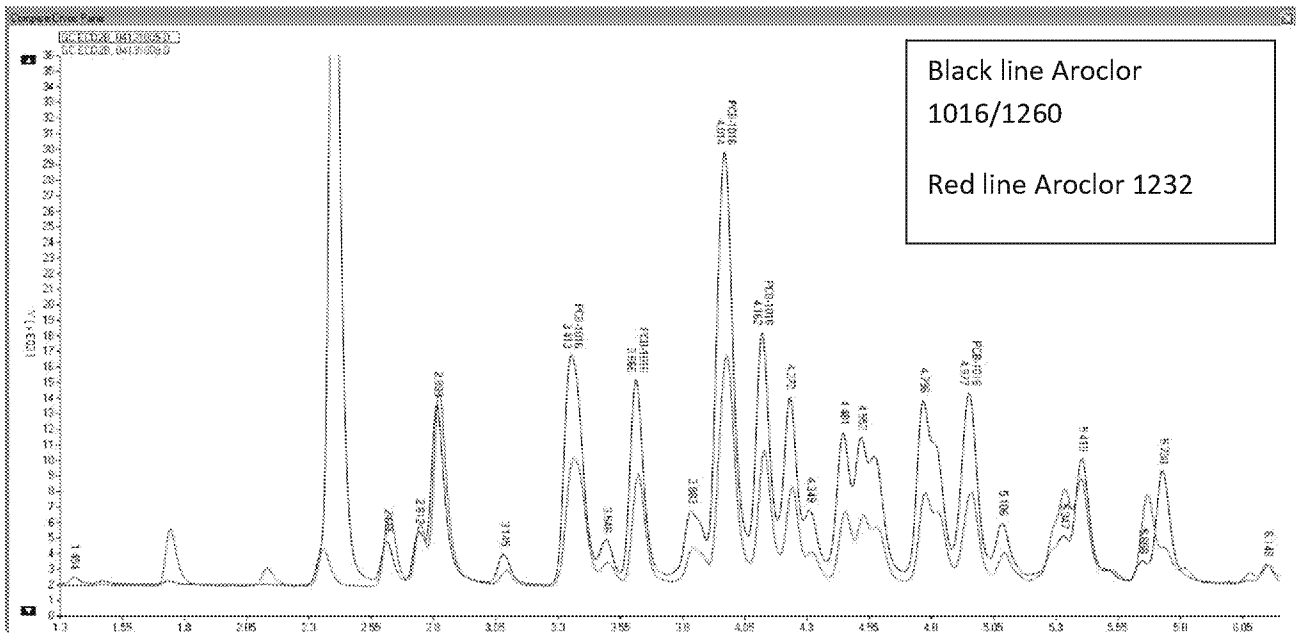
The first example considers Aroclor 1016 vs. 1242. Note that the early part of the chromatograms (2.5 – 5.3 minutes) are virtually identical. The key difference is the presence of some later eluting peaks (6.2-7.3 minutes in this chromatogram) in 1242 that are not present in 1016. This difference is masked by the fact that Aroclor 1260 is also present in this standard. Most labs analyze standards of 1016 and 1260 together – there is nothing wrong with this but it is a good idea to periodically (one run with each initial calibration?) analyze them separately so that you have a good idea of the two separate patterns.

The story of 1016 is interesting – in the early 1970's PCBs were starting to be found in fish in the Great Lakes. The more heavily chlorinated biphenyls were bioaccumulating more and were of greatest concern. So, Monsanto attempted to modify the manufacturing process to reduce the amount of pentachlorobiphenyls in Aroclor 1242, while still keeping the overall degree of chlorination similar. They were successful in this regard – Aroclor 1242 has about 10% pentachlorobiphenyls which show up between 6 and 7.4 minutes in the chromatogram below. Aroclor 1016 has 42% by weight chlorine (it does not follow the standard naming convention) but has no pentachlorobiphenyls.

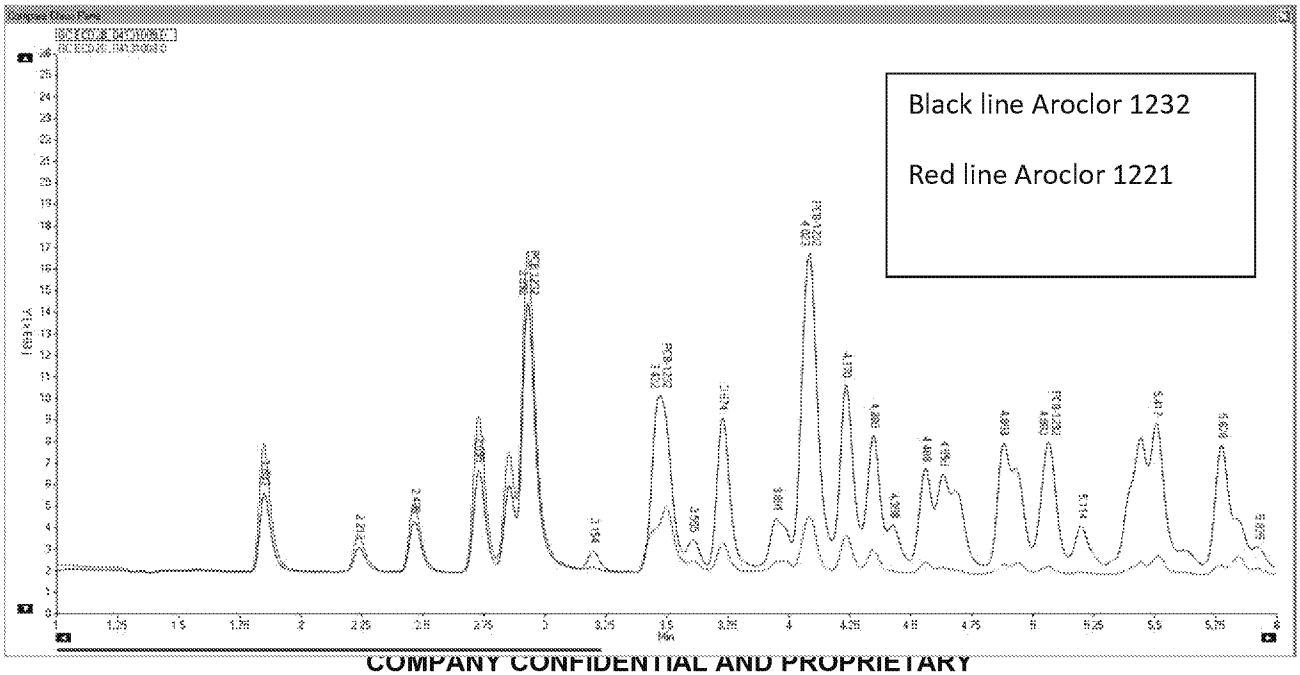


Attachment 1. Aroclor Identification 101 (Continued)

Here is 1016 vs. 1232. These are even more similar (the large peak at around 2.35 min is TCMX) but note the very early peaks present in 1232 and not in 1016, and also note that the front end is stronger in 1232 for example in 1232 the peak at 2.88 min is about the same size as those at 4.79 and 4.97, whereas in 1016 the later peaks are twice as large.



For 1221 vs. 1232, the front end of the chromatogram is identical, but 1232 has later peaks that are not present in 1221.



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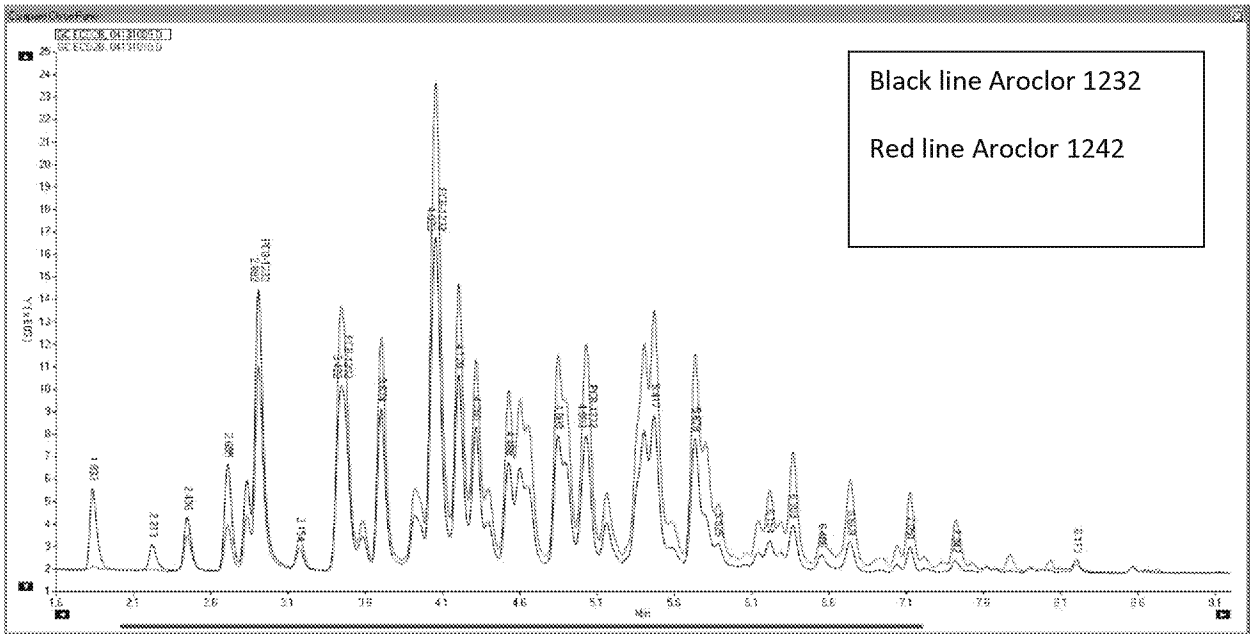
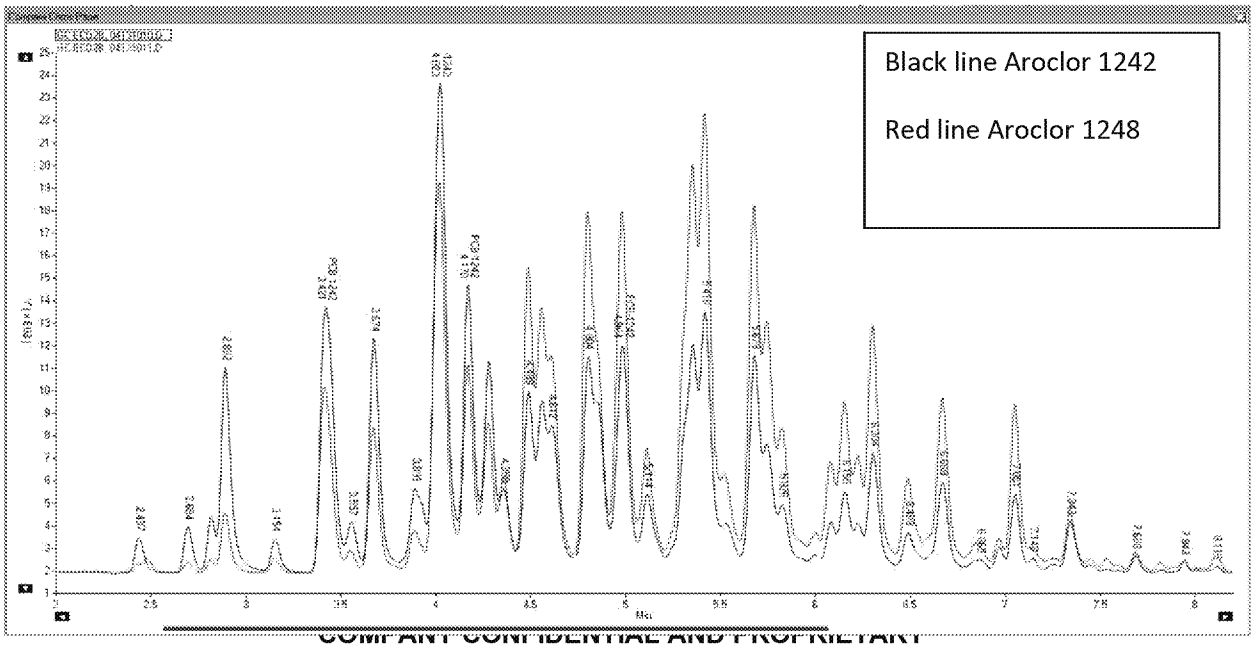
Attachment 1. Aroclor Identification 101 (Continued)

1232 and 1242 are best distinguished by the early peaks in 1232 (1.83, 2.13) that are not present in 1242. Also note that the peak at 2.89 is twice the height of that at 5.41 in 1232, whereas the 5.41 peak is slightly higher in 1242. This relative size of the front and back end of the envelope is a key tool for distinguishing Aroclors.



Aroclors 1242 and 1248 both have all of the same peaks, so the relative strength of the front and back of the envelope is the only way to distinguish. For example, in 1242, the peak at 3.42 is larger than that at 5.67, whereas for 1248, the 5.67 peak is considerably larger than the 3.42 peak.



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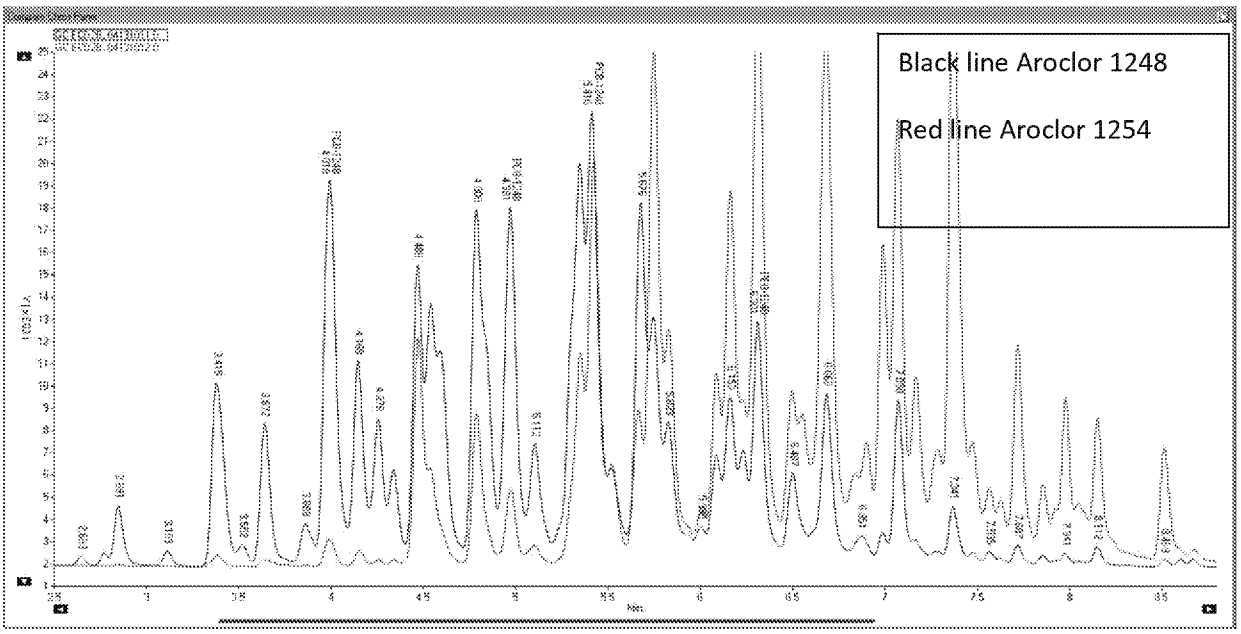
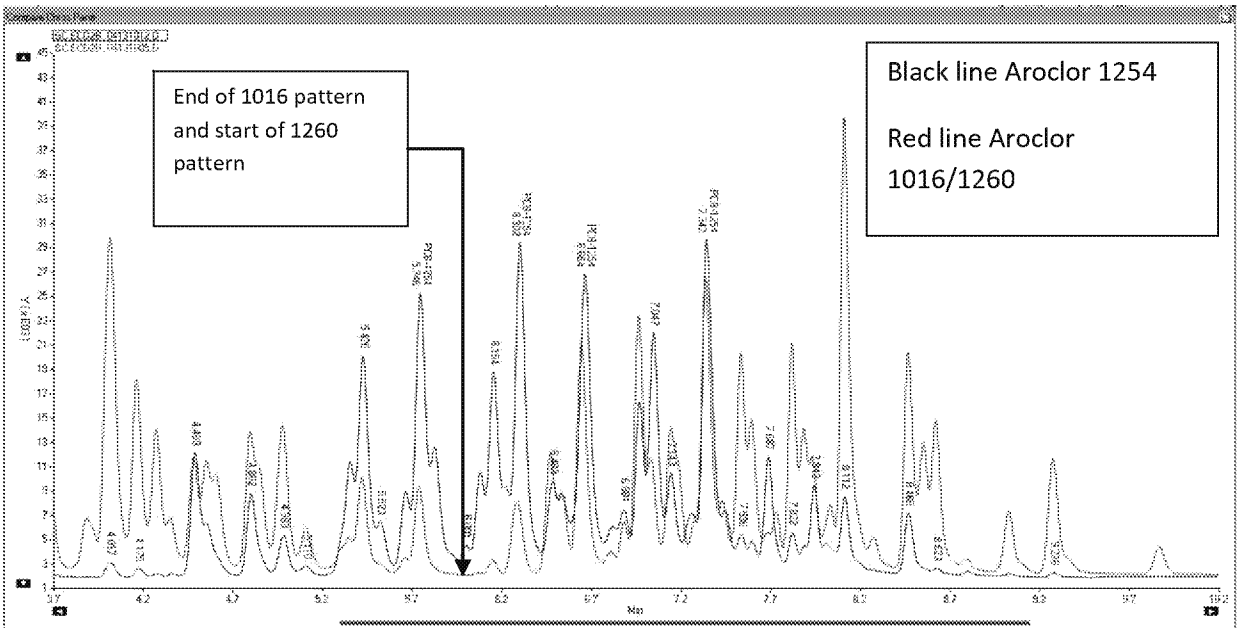
Attachment 1. Aroclor Identification 101 (Continued)

1248 vs 1254 is a relatively easy case, the front end of the envelope is much stronger in 1248.

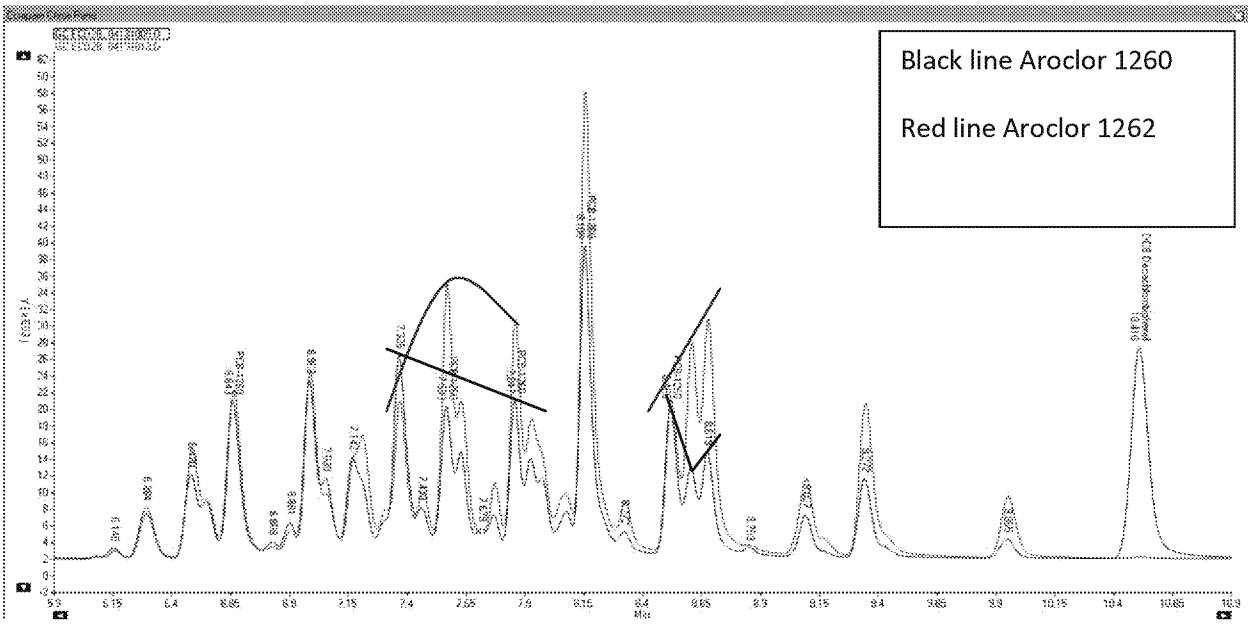


The 1254 vs. 1260 chromatograms are again a little masked by the inclusion of 1016 in the 1260 standard (peaks up to 5.9 min in the 1260 chromatogram actually belong to 1016). Keeping this in mind, the presence of peaks at 5.42 and 5.74 indicates 1254. The relative strength of peaks in the 7.5-9.3 range indicates 1260.

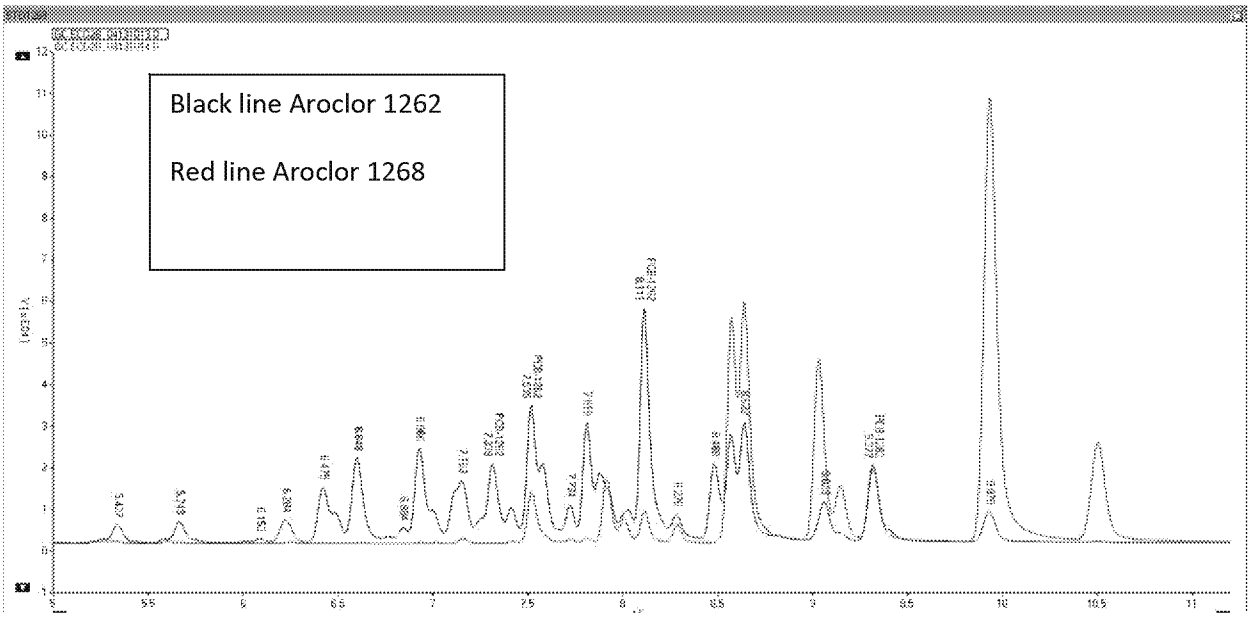


Attachment 1. Aroclor Identification 101 (Continued)

Many labs do not analyze for 1262, perhaps just as well since it is certainly challenging to distinguish from 1260. However, the shape of the envelope is again the key. Note that in 1262 the peaks around 8.6 minutes are as large as that at 6.96, whereas they are only half the size in 1260. Also note the shape of the envelope for the peaks in the 7-8 minute range – bow shaped for 1262 and a straight declining line for 1260. The envelope shape is also quite different in the 8.4-8.7minute range.

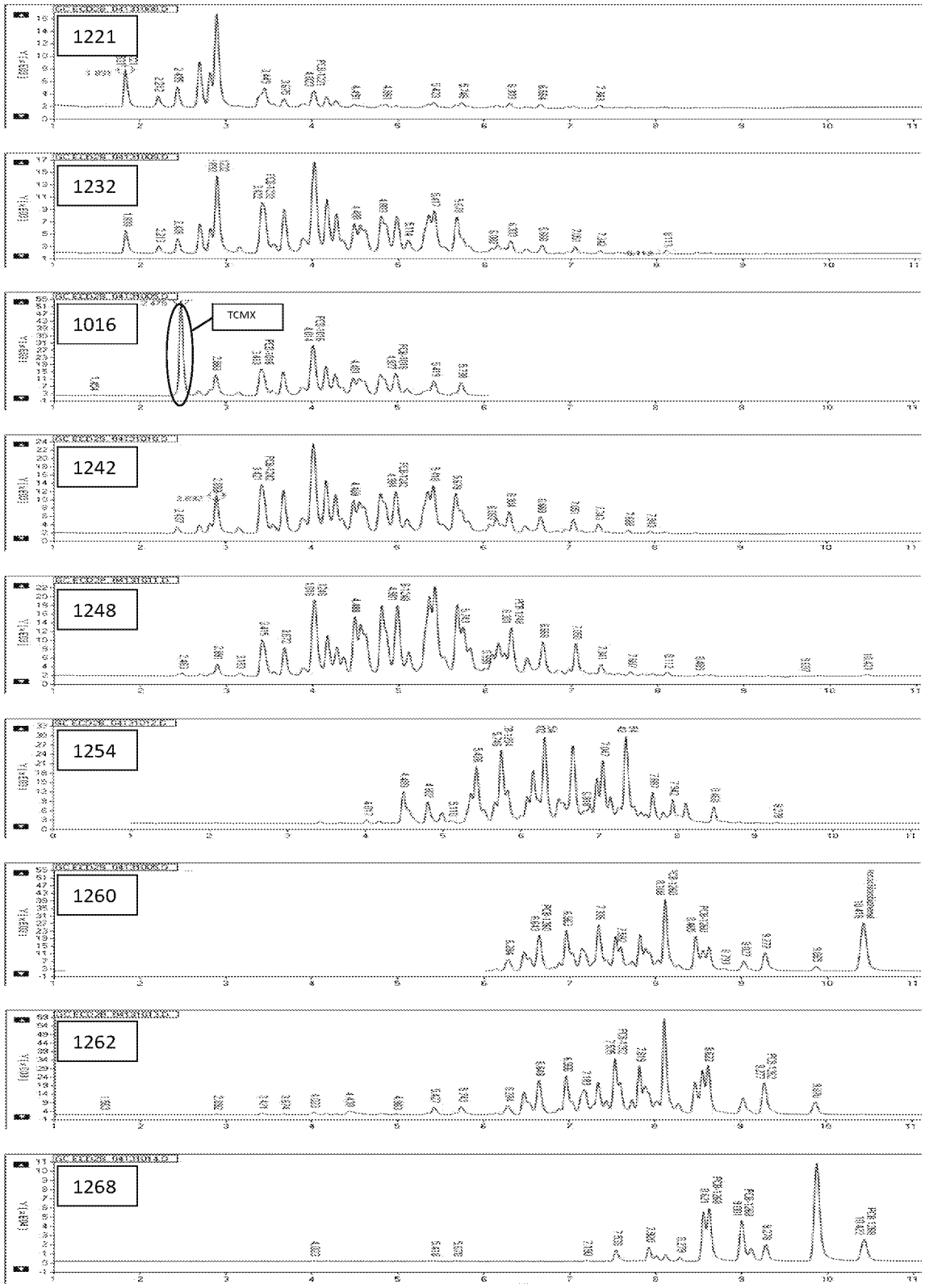


The really strong peak at 9.87 and the lack of much of a pattern between 6.0 and 7.5 minutes are good indicators of 1268.



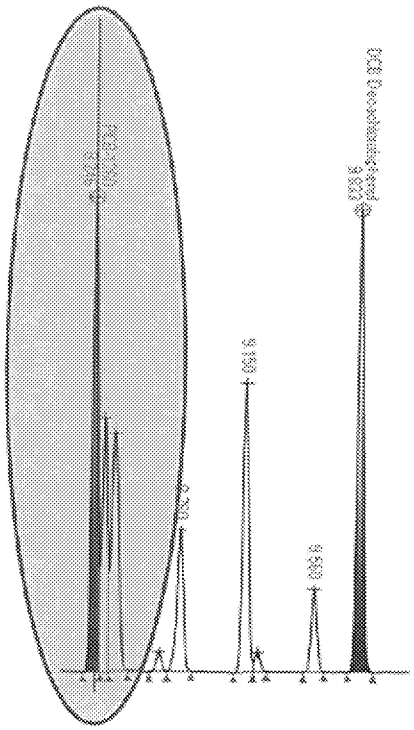
Attachment 1. Aroclor Identification 101 (Continued)

AROCLOR PATTERNS



Attachment 2.
Example of Minimum Resolution Requirement for Aroclor 1260 Triplet

(See Work Instruction CA-T-WI-003 for more information)



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

Work Instruction No. CA-T-WI-003, dated 31 Mar 2015

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Electronic Copy Only

Title: Diesel and Residual Range Organics (DRO and RRO) by GC/FID

[SW846 Method 8015 and State Methods]

Approvals (Signature/Date):

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3/3/20	3/3/20
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1.0 Scope and Application

1.1 This procedure is designed to measure the concentration of diesel range organics (DRO), jet fuels, and motor oils in water or solid samples by EPA Methods 8015B, 8015C, 8015D, using method 8000B and 8000C criteria. Also addressed in this SOP are Alaska Methods AK102 and AK103, and the NWTPH (Washington). The Oklahoma method appears in the appendix.

1.2 As most commonly defined, extractable hydrocarbons corresponds to a carbon range of $C_{10} - C_{40}$ (boiling point range of approximately 170 °C to 560 °C). This carbon range can include kerosene, several types of jet fuel, several types of diesel fuels, and a number of types of light heating oils. Other petroleum hydrocarbon ranges, any within the $C_8 - C_{40}$ general range, may be characterized using this general method (details concerning the calibration and aliphatic reference standards need to be specified in the project work plan, and the final report must clearly indicate the range used).

1.3 Table 1 provides boiling point information for the aliphatic hydrocarbons.

1.4 Petroleum products such as lubricating oils, waxes, and asphalt can have a significant fraction of higher molecular weight / higher boiling point components that are not detectable under the conditions of this method.

1.5 Analytes, Matrix(s), and Reporting Limits

1.5.1 The standard diesel reporting limit for this procedure is 0.25 mg/L for water samples and 4.0 mg/kg for soil samples. The jet fuel and motor oil reporting limits are 1 mg/L (0.5 mg/L for NWTPH) for water samples and 12 mg/kg for soil samples. Other reporting limits may apply to each of the listed methods with the level 1 initial calibration standard being the basis of the calibration for the lowest reporting limit.

1.5.2 Unless otherwise noted the most stringent criteria or common criteria will be reported and adhered to for the methods listed above.

1.5.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in SOP DV-QA-0031.

2.0 Summary of Method

2.1 The method involves extracting hydrocarbons from aqueous or solid samples with methylene chloride. See SOPs DV-OP-0006, DV-OP-0016, DV-OP-0015, and DV-OP-0007 for water sample extraction, solid sample extraction, and extract concentration procedures, respectively. For solvent-miscible products, medium/high level options are provided. All associated methods typically extract 1 L of water to a final volume of 1 mL. For method 8015 the typical soil amount is 30 g. The laboratory typically extracts 30 g of soil by 3550, or 15 g of soil by 3546.

Method AK102 calls for the extraction of 10-30 g of soil and NWTPH indicates that 20 g of soil is extracted. The laboratory typically extracts 30 g of soil for AK102 and NWTPH. All soil extractions are taken to a final volume of 1 mL if possible.

2.2 The methylene chloride extract is concentrated and analyzed with a gas chromatograph equipped with a flame ionization detector (GC/FID). Quantitation is performed by comparing the total peak area within the specified carbon range, both resolved and unresolved peaks, to the response of an authentic fuel or oil calibration standard.

3.0 Definitions

3.1 Diesel Range Organics (DRO): The sum of compounds producing chromatographic peaks, both resolved and unresolved, eluting from:

- the start of the n-decane peak (C_{10}) to the end of n-octacosane (C_{28}), or
- n-decane (C_{10}) to (beginning of for AK102) n-pentacosane (C_{25}), or
- n-decane (C_{10}) to n-dotriacontane (C_{32}), or
- n-decane (C_{10}) and n-hexatriacontane (C_{36}) for extended DRO.

The markers used for integration for quantitation of DRO varies by method. In order for the quantification to be effective the other methods should likewise include the entire hydrocarbon range for the representative hydrocarbon standard material in the calibration. It is not recommended that narrower carbon ranges be selected compared to the wider ranges available in the standards.

Method	Quantitation Markers
8015	Integration begins at the apex minus the RT window for the starting alkane and ends at the apex plus the RT window for the ending alkane Marker
Oklahoma	Integration begins 0.1 minute before the initial alkane and ends 0.1 minutes beyond the ending alkane marker.
NWTPH	Integration includes the whole of the hydrocarbon pattern (peaks at least down to 10% of the highest representative peak) and that the integration breaks where multiple hydrocarbon patterns are indicated for a single sample.

3.2 Diesel Calibration Standard: A retail-purchased diesel #2 or fuel oil #2 (the two products are essentially the same crude oil distillation cut) used as the calibration stock. The Oklahoma standard uses a ten component (C10-C28, even homologs) blend of alkanes.

3.3 Motor Oil (MO): The sum of the compounds producing chromatographic peaks, both resolved and unresolved, eluting from

- the start of the n-cosane (C_{20}) to n-octatriacontane (C_{38}), or

- (the beginning of for AK103) n-pentacosane (C_{25}) to (the end of for AK103) n-hexatriacontane (C_{36}).

Alaska Method AK103 defines this latter range as "residual range organics" or RRO.

3.4 Jet Propellant-8 (JP-8): Also referred to as Jet fuel-8. The hydrocarbon range for this fuel is typically from C_8 to C_{12} or from the start of n-octane to the end of n-dodecane. In cases where the solvent peak interferes with the integration of the C_8 part of the chromatogram an adjustment may be made to start the integration just past the end of the solvent front.

3.5 Oklahoma: These carbon ranges and standards are defined and explained in detail in Appendix 1.

3.6 Alaska Method AK102: Method for the determination of diesel range organics defines DRO as a carbon range from the beginning of C_{10} to the beginning of C_{25} (boiling point range of approximately 170°C to 400°C).

3.7 Alaska method AK103: Method for the determination of residual range organics (RRO) defines RRO as a carbon range from the beginning of C_{25} to the end of C_{36} (boiling point range of approximately 400°C to 500°C). It should be noted that this is essentially the same range as defined for motor oil.

4.0 Interferences

4.1 Although primarily designed as a petroleum hydrocarbon test procedure, other organic compounds including, but not limited to, animal and vegetable oil and grease, chlorinated hydrocarbons, phenols, phthalate esters, and biogenic terpenes are measurable under the conditions of this method. A silica gel cleanup can be used to remove these biogenic and substituted hydrocarbons. The details of the cleanup procedure are found in SOP DV-OP-0007.

4.2 Heavier petroleum products such as lubricating oil and crude oil also produce a response within the retention time range for DRO. As defined in the method, the DRO results include these compounds.

4.3 Method interferences are reduced by washing all glassware in accordance with SOP DV-OP-0004. At least one method blank must be analyzed with each extraction batch to demonstrate that the samples are free from method interferences.

4.4 High-purity reagents must be used to minimize interference problems.

4.5 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a solvent blank to check for cross-contamination. It is the laboratory's policy to reanalyze any samples that follow an unusually concentrated sample (above the high level calibration

standard) and that show detectable levels of the same compounds that appeared in the preceding concentrated sample.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 Specific Safety Concerns or Requirements

5.2.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.2.2 The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of these zones, and must cool them to room temperature prior to working on them.

5.2.3 There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and Symptoms of Exposure
Methylene Chloride	Carcinogen Irritant Poison	25 ppm – TWA 125 ppm – STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting, and headache. Causes irritation, redness, and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm – TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Silica Gel	No physical or Health Hazards under GHS	Not Determined	May cause Irritation, shortness of breath, headache, Nausea, Dizziness if inhaled, skin and/or eye contact or swallowed.
(1) Always add acid to water to prevent violent reactions. (2) Exposure limit refers to the OSHA regulatory exposure limit..			

6.0 Equipment and Supplies

6.1 Instrumentation

Gas Chromatograph: Analytical system including appropriate gas supply and all required accessories, including a Flame Ionization Detector (FID), column supplies, gases, and syringes. A data system capable of determining peak areas using a forced baseline and baseline projection is required. A data system capable of storing and reintegrating chromatographic data is recommended. The instruments used for this SOP are Hewlett Packard 5890 series II instrument U and Agilent 7890 instrument U2.

6.2 Supplies

6.2.1 Chromatography Columns

6.2.1.1 Restek Corporation Rtx®-1 (fused silica), 30 m, 0.32 mm ID, 0.25 µm film thickness used for both U and U2 .

6.2.1.2 Other columns that meet the performance criteria stipulated in Section 10.0 may be used. Capillary columns are required to achieve the necessary resolution.

6.2.2 2-mL glass vials with Teflon-lined cap (autosampler vials) and limited volume liners.

6.2.3 Thermogreen septa, ferrules, Siltek injection port liners, Siltek glass wool. Use Agilent liner 5183-4647 for instrument U2 and use Goosneneck Splitless Resteck Liner 20799 with Restek deactivated wool 24324 for U.

6.2.4 Microsyringes: 1, 5, 10, 25, 100, 250, 500, 1000, and 5000 µL.

6.2.5 Volumetric flasks: 10.0, 50.0, and 100 mL, Class A glass.

6.2.6 An analytical balance capable of accurately weighing 0.0001 g must be used for preparing standards. A top-loading balance capable of weighing to the nearest 0.01 g must be used for sample preparation.

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Methylene chloride, pesticide grade or equivalent. Each lot must be approved for use in accordance with CA-Q-S-001.

7.1.2 Acetone, pesticide grade or equivalent. Each lot must be approved for use in accordance with CA-Q-S-001.

7.1.3 Ottawa sand (reagent sand) for solid matrix method blanks and lab control samples.

7.1.4 Carrier gas, >/= 99.999999% pure hydrogen.

7.1.5 Make-up gas, >/= 99.99980% pure nitrogen.

7.1.6 Compressed air for the FID.

7.2 Standards

NOTE: The availability of the specific commercial standard solutions upon which the following sections are based may change at any time. As a result, it may be necessary to alter the dilution scheme presented herein to accommodate changes in stock standard concentrations. All such changes are documented in the standards preparation records.

NOTE: When logging stock standards into the reagent module of TALS, the concentration of the DRO and RRO ranges are determined as outlined below. These percentages were determined by calculating the ratio of the area count of the narrower ranges to that of the largest range.

DRO: C10-C36 is the largest reportable range and is therefore logged with a concentration equivalent to the total DRO concentration of the standard. The concentration of C10-C32 equals 99.6% of the total DRO concentration of the standard. The concentration of C10-C28 is logged as 99.2% of the total DRO concentration of the standard. The concentration of

C10-C25 is logged as 98.1% of the total DRO concentration in the standard.

RRO: C20-C38 is the largest reportable range and is therefore logged with a concentration equivalent to the total motor oil concentration of the standard. The concentration of C25-C36 equals 75.0% of the total motor oil concentration of the standard.

7.2.1 RT Reference Standard: **(DRO_RTMKR1 and DRO_RTMKR2)** DRO Mixture (Tennessee/Mississippi) and Calibration/Window Defining Hydrocarbon Standard. Current source Accu Std DRH-004S-R1-5x and Ultra Sci UST-210 both at 1000 ug/ml.

7.2.2 Stock Diesel Fuel Standard: **(DRO_Fuel2)** – Vendor purchased Diesel fuel oil #2 at a concentration of 50,000 µg/mL in methylene chloride. Current source Restek 31259.

7.2.3 Stock Motor Oil / Residual Range Organics (RRO) Standard: **(RRO_Comp)** – Equal weights of 30-weight and 40-weight motor oils are composited and used to make this standard. Alternatively, the Alaska method standard may be purchased from a commercial source as a stock solution in methylene chloride at a concentration of 50,000 µg/mL. Current source is Ultra Sci RGO-724.

7.2.4 Stock Jet Fuel Standards, JP8: **(DRO_JP8)** - Purchased from a vendor at concentrations of 50,000 µg/mL in methylene chloride. Current source is Restek 31254.

7.2.5 Stock MRH/HRH Standard: **(KS_DRO_Primar)** – Vendor purchased Aliphatic hydrocarbon standard at a concentration of 6,000/8,000 µg/mL in hexane. Current source – Ultra SMA-310.

7.2.6 Stock n-Pentatriacontane Standard: **(KS_DRO_C35)** – Vendor purchased n-Pentatriacontane standard at a concentration of 1,000 µg/mL in hexane. Current source – SPEX S-2990.

7.2.7 Stock Surrogate Solution: **(DRO_SURR)** – Purchased as a custom mix from a vendor, consisting of ortho-terphenyl and n-octacosane, at a concentration of 5,000 µg/mL each in methylene chloride. Accu Std S-8087-2.5x.

NOTE: Alaska Method AK103 (RRO) requires the use of n-triacontane-d_{62} as a surrogate. However, the Alaska Certification Office has granted approval to use n-octacosane (C_{28}) as the surrogate.

7.2.8 Surrogate Spike Solution: **(8015SurStock)**– Refer to SOP DV-OP-0020

7.2.9 LCS Spike Solution: (8015LCS) (8015RROLCS) – Refer to SOP DV-OP-0020 for instructions and table

7.2.10 Diesel Fuel Calibration Standards: **(DRO L#)**– Diesel fuel calibration standards are prepared in methylene chloride at 7 concentration levels using either the Stock Diesel Fuel Standard (Section 7.2.2) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Diesel Conc (µg/mL)	Surrogate Conc (µg/mL)
7	Stock	0.6	1.0	30,000	NA
6	Stock + Stock Surrogate	1.5 and 0.075	5.0	15,000	75.0
5	Level 6	0.5	1.0	7500	37.5
4*	Stock + Stock Surrogate	1 and 0.05	10	5000	25.0
3	Level 4	0.2	1.0	1000	5.0
2	Level 4	0.05	1.0	250	1.25
1	Level 4	0.02	1.0	100	0.5

NOTE: The Level 4 (*) calibration standard is also used as the continuing calibration verification (CCV) standard. Only the diesel fuel calibration standards contain the surrogates.

7.2.11 Jet Fuel Calibration Standards: **(DRO_JP8_L#)**– Jet Fuel (JF) Calibration standards are prepared in methylene chloride at 6 concentration levels using either the Stock Jet Fuel Standards (Section 7.2.4) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc (µg/mL)
6 JF	Stock	1	5	10,000
5 JF	Level 6 JF	0.125	0.250	5000
4 JF	Level 6 JF	0.0625	0.250	2500
3 JF*	Stock	0.2	10.0	1000
2 JF	Level 3 JF	0.125	0.250	500
1 JF	Level 3 JF	0.025	0.250	100

NOTE: The Level 3 JF (*) jet fuel calibration standard is used as the CCV standard.

7.2.12 Motor Oil / RRO Calibration Standards: **(RRO_L#)** Motor Oil (MO) (or Alaska Method RRO) calibration standards are prepared in methylene chloride at 7 concentration levels using either the Stock Motor Oil / RRO

Standard (Section 7.2.3) or a higher level calibration standard as summarized in the following table:

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc (µg/mL)
7RRO	Stock	0.6	1.0	30,000
6RRO	7RRO	0.5	1.0	15,000
5 RRO	7RRO	0.250	1.0	7500
4 RRO*	Stock	0.5	5.0	5000
3 RRO	Level 4 RRO	0.2	1.0	1000
2 RRO	Level 4 RRO	0.05	1.0	250
1 RRO	Level 4 RRO	0.02	1.0	100

NOTE: The Level 4 RRO (*) calibration standard is used as the CCV standard.

7.2.13 Diesel Fuel Continuing Calibration Verification Standard (CCV): The Level 4 Diesel Fuel Calibration Standard (Section 7.2.8) is used as the CCV standard.

7.2.14 Jet Fuel CCV Standard: The Level 3 JF jet fuel calibration standard (Section 7.2.11) is used as the jet fuel CCV standard.

7.2.15 Motor Oil / RRO CCV Standard: The Level 4 MO motor oil / RRO calibration standard (Section 7.2.12) is used as the motor oil / RRO CCV standard.

7.2.16 Second-Source Calibration Verification Standards: A standard containing diesel fuel in methylene chloride at a concentration of 2,000-µg/mL or motor oil/RRO in methylene chloride at a concentration of 2,500-µg/mL and jet fuel at a concentration of 1,000 □g/mL is used to verify each initial calibration. This standard uses diesel fuel, jet fuel and motor oil obtained from a source different than the source for the primary standard.

(DRO_SS) AccuStandard DRO-AK-102_LCS-10x at 50 mg/mL. Stock surrogate standard. The second source is prepared by diluting 1 mL of DRO and 100 µL of surrogate to a final volume of 25 mL with methylene chloride.

(RRO_SS) AccuStandard FU-026-D-40x (30 weight) and FU-027-D-40x (40 weight) each at 20 mg/mL are combined in equal portions. The second source is prepared by diluting 625 µL of each stock to a final volume of 10 mL in methylene chloride.

(8015_JP8_SS) AccuStandard FU-015-D-40x at 20 mg/mL. The second source is prepared by diluting 250 µL to a final volume of 5 mL in methylene chloride.

7.3 Standards Verification

The standards verification process is described in SOP DV-QA-0015.

7.4 Storage of Stock Standards

Stock standard solutions are stored per vendor recommendations, generally at room temperature. Working level standard solutions are kept refrigerated at ≤ 6 ºC. Standard solutions must be replaced every six months or sooner as specified by the vendor. Standard solutions must be replaced more frequently if comparison with check standards indicates a problem.

7.5 Non-Routine Compounds

Other, non-routine compounds not listed in this section may be requested by a client and may be added to this procedure.

7.5.1 In these cases, all stock solutions will be obtained from commercial sources and will be verified with a second-source standard as described in SOP DV-QA-0015.

7.5.2 Non-routine standards will be stored and treated as described in Section 7.4 above or as specified by the manufacturer.

7.5.3 Subsequent dilutions of specially requested compounds will be determined in a manner consistent with the client's recommendations for number of calibration points, inclusion of reporting limit, and concentration range adequate to represent the linearity of the instrument.

7.5.4 These specially requested, non-routine compounds either may be added to the dilution scheme used for routine compounds or may be prepared as a separate calibration.

7.5.5 All standards preparation for non-routine compounds shall be documented using the same method that is used for routine compounds.

7.5.6 The carbon range must be specified to accommodate the entirety of the hydrocarbon pattern.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Water	2 x 1 liter amber	1 liter	Cool, ≤6°C and not frozen	7 Days	40 Days from extraction	SW-846
Water[1]	2 x 1 liter amber[1]	1 liter	Cool, ≤6°C and not frozen pH < 2, HCl	14 Days	40 Days from extraction	AK102/103 NWTPH

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Soil	4- 8 oz amber Jar	30 g by 3550 15 g by 3546	Cool, ≤6°C and not frozen	14 Days	40 Days from extraction	SW-846 AK102/103 NWTPH

[1] If requesting MS/MSD for AK102/103, must send at least 5 x 1 L bottles because method requires MS/MSD for DRO and RRO, which can't be spiked together.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.1 unless otherwise stated. Any deviation or exceptions from QSM 5.1 requirements must have prior approval in the project requirements. DoD QSM requirements and standard variances (if approved) for this procedure are summarized in Table VII.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Initial Demonstration of Capability

Before analyzing samples, the analyst must demonstrate the ability to operate the instrumentation and generate data that meets method criteria (IDOC). The analyst must also establish a method detection limit (MDL) for each instrument used for a particular method of analysis. The initial demonstration and method detection limit

(MDL) studies (see Policy DV-QA-005P) must be acceptable before analysis of samples may begin. An MDL verification standard prepared at approximately two times the MDL is analyzed quarterly to demonstrate the qualitative reliability of the calculated MDL value. MDL studies are performed when major changes are made to the analytical processes. MDLs are stored in the LIMS. Likewise ongoing proficiency must be demonstrated by each analyst on an annual basis. See Section 13 for more details on detection limit studies, initial demonstrations of capability, and analyst training and qualification.

9.3 Sample QC

9.3.1 Quality Control Batch

The batch is a set of up to 20 samples of the same matrix processed together using the same reagents and standards. Each quality control batch must contain a method blank (MB), a laboratory control sample (LCS), and matrix spike - matrix spike duplicate (MS/MSD) pair. If there is not enough sample volume for an MS/MSD then a duplicate LCS may be used to determine precision. For more details see Policy DV-QA-003P.

NOTE: The AK-102 and AK-103 Methods require duplicate LCSs (i.e., LCS and LCSD) for each batch. If both ranges are requested, two sets of LCS/LCSD are required, one for each range. The NWTPH and Oklahoma methods specify that duplicates be supplied every 10 samples.

9.3.2 Method Blank (MB)

One method blank is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The method blank consists of either 1 liter of organic-free water (for batches of aqueous samples) or 30 grams of Ottawa sand (for batches of soil samples by 3550) or 15 grams of Ottawa sand (for batches of soil samples by 3546. The method blank is processed exactly as samples in the batch, and is used to assess whether the laboratory processes have contaminated the samples in the batch.

Acceptance Criteria: Results for the method blank must be less than or equal to one-half the reporting limit concentration or less than 10% of the lowest concentration found in the associated samples. For DoD QSM 4.2 or QSM 5.1 the acceptance criteria is no analytes detected >1/2 RL (i.e., LOQ) or >1/10 the amount measured in any sample or 1/10 the regulatory limit whichever is greater.

Corrective Action: If the method blank acceptance criteria are not met, identify and correct the source of contamination, and re-prepare and reanalyze the associated samples. If the analyte that was present

in the MB was not detected in the affected samples, then the data may be reported with qualifiers (check the project's requirements to be sure that this is allowed) and it must be addressed in the projects narrative.

9.3.3 Laboratory Control Sample (LCS)

9.3.3.1 With the exception of sample batches analyzed by Methods AK-102 or AK-103, one LCS is analyzed with every preparation batch or every 20 samples, whichever is more frequent. The LCS consists of either 1 liter of organic-free water (for batches of aqueous samples) or 30 grams of Ottawa sand (or method designated sample size as noted in Section 9.3.2 for batches of soil samples), to which the analytes of interest are added at a known concentration.

9.3.3.2 Methods AK-102 and AK-103 require the analysis of an LCS and a duplicate LCS (LCSD) with each preparation batch or every 20 samples, whichever is more frequent. The LCS and LCSD consists of either 1 liter of organic-free water (for batches of aqueous samples) or 25 grams of Ottawa sand (for batches of soil samples), to which the analytes of interest are added at a known concentration.

9.3.3.3 See Table III for spike levels and section 7.2.9 for preparation of the spiking solution. The LCS is processed exactly as samples in the batch and is used to assess the accuracy of the analytical system.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. For Alaska Methods AK102 and AK103, the acceptance limits are listed in Table V. Oklahoma method specifies a recovery of +/- 20% (80-120%) for waters and +/- 40% (60-140%) for soils. For all other methods, the control limits are set at ± 3 standard deviations around the calculated mean of the historical LCS recovery data, unless project-specific control limits apply. DoD QSM 5.0 limits apply to projects under this document. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

Corrective Action: If LCS acceptance limits are not met, check the analytical system for proper function and calibration control, make any corrections, then the LCS should be reanalyzed once to confirm that the original analysis is reliable. If

the results are still outside control limits, the extraction process must be examined and corrected where problems are identified. The associated samples must then be re-extracted following the corrected procedure and reanalyzed. If the LCS recovery is above the upper control limit, and the associated samples are all below reportable concentrations, the deviation may be described in an NCM, if this is acceptable to the client or allowed by the specific program or project.

9.3.4 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

One matrix spike (MS) and one matrix spike duplicate (MSD) are prepared for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. It is prepared in a manner similar to the LCS, but uses real sample matrix in place of the blank matrix. Field blanks and equipment rinses must not be used to prepare the MS and MSD. The MS and MSD are processed exactly as samples in the batch, and are used to assess the effects of sample matrix on the accuracy and precision of the analytical system. See Table III for spike levels and see section 7.2.7 for preparation of the spiking solution. For the DoD QSM 4.2 or QSM 5.0, the MS/MSD must be from the project site and insufficient sample is available to analyze the MS/MSD pair, this is documented in an NCM and an LCSD is performed.

Acceptance Criteria: The percent recovery of the analytes of interest must fall within the established control limits. The control limits are set at ± 3 standard deviations around the calculated mean of the historical MS recovery data, unless project-specific control limits applyDoD QSM 5.0 limits apply to projects under this document for Method 8015. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

The relative percent difference (RPD) between the MS and MSD must be less than the established control limit, which is based on 3 standard deviations of the mean of the historical data. RPD control limits are maintained in the laboratory LIMS.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to

accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not

the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE 1: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE 2: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

9.3.5 Surrogate Spikes

The calibration standards, field samples, and QC samples are spiked with ortho-terphenyl and n-octacosane as surrogates. These surrogates have chemistry similar to the analytes of interest, but are not expected to be found in environmental samples. See Table III for spike levels and Sections 7.2.5 and 7.2.6 for surrogate spike preparation. Surrogate results are used to assess the performance of the analytical system for each field and QC sample.

Acceptance Criteria: The percent recovery of the surrogates must fall within the established control limits. For Alaska Methods AK102 and AK103, the acceptance limits are listed in Table V. For DoD QSM 5.0, QSM limits apply for Method 8015. For all other methods, the control limits are set at ± 3 standard deviations around the calculated mean of the historical surrogate recovery data, unless project-specific control limits apply. Current control limits are stored in the laboratory LIMS. See Policy DV-QA-003P for further details.

Corrective Action: If surrogate recoveries in the method blank are outside the established limits, verify calculations, standard solutions, and acceptable instrument performance. High surrogate recoveries in the blank might be acceptable if the surrogate recoveries for the field samples and other QC samples in the batch are in control. Low surrogate recoveries in the blank require re-preparation and

re-analysis of the samples, unless the sample surrogate recoveries are in control and the targeted compounds are not found in the affected samples.

If the sample surrogate recoveries are outside the established limits, verify calculations, dilutions, standard solutions, and acceptable instrument performance. High recoveries may be due to a co-eluting matrix interference and the chromatogram should be examined for evidence of this. Low recoveries may be due to adsorption by the sample matrix (e.g., clay particles, peat, or organic material in the sample). Recalculate the results and/or reanalyze the extract if the checks reveal a problem.

If the surrogate recovery is outside the established limits due to well-documented matrix effects, the results must be flagged and an explanation included in the report narrative. As with matrix spike failures, some programs may require additional analyses to confirm suspected matrix interferences. The decision to reanalyze or flag the data should be made in consultation with the client. If matrix interference is not obvious, it is only necessary to re-prepare / reanalyze a sample once to demonstrate that a matrix effect is reproducible. The decision to re-prepare samples for out of control surrogate outside of the holding time should be made in consultation with the client.

9.3.6 RT Reference Standard

A combination of two n-alkane mixtures is analyzed to establish the retention time (RT) window for each initial calibration. The Tennessee / Mississippi DRO mixture is composed of all n-alkanes from C_{10} – C_{25} and the Calibration / Window Defining Hydrocarbon Standard is composed of all even-numbered n-alkanes from C_8 – C_{40}. The instrument conditions should be chosen so that each alkane component is completely resolved from the next alkane component (this is necessary in order to define each range as different from another specified range). The RT standard data must be uploaded into each analytical batch for the applicable carbon range as the RTC.

10.0 Calibration and Standardization

10.1 Instrumentation

10.1.1 TestAmerica Denver gas chromatograph instrument systems are computer controlled to automatically inject samples and process the resulting data.

10.1.2 Instrument conditions are shown in Table IV.

10.1.3 The routine injection volume is 1 µL.

10.1.4 GC run conditions and columns must be chosen to meet the acceptance criteria for the RT Reference Standard listed in Section 9.3.6 and the calibration criteria in Section 10.

10.1.5 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and under the public folder Arizona Calibration Training.

~~10.1.6~~ Unprocessed calibration data are transferred to the Chrom database for processing. After processing the calibration data, upload calibration to TALS. Lock down the calibration in TALs and acknowledge the Data Review Checklist (DRC). Lock all limit groups down in Chrom. Submit Calibration batches for 2nd level review

10.1.7 A new calibration curve must be generated initially, after major changes to the system, when continuing calibration criteria cannot be met, or at least annually. Major changes include installation of new columns and changing FID jets.

10.2 Calibration

10.2.1 The ICAL is performed using the concentration levels described in Sections 7.2.8 for diesel fuel, 7.2.11 for the jet fuels, and 7.2.12 for motor oil. Calibration levels are also presented in Table II. Although some methods allow as few as three calibration levels, the minimum number of calibration levels will be maintained as five levels.

10.2.1.1 Several separate initial calibration curves (ICALs) are required to calibrate for all the mixtures. An ICAL must always be maintained for the diesel fuel as these standards contain the surrogate compounds. ICALs for the other mixtures are analyzed as needed, depending upon the requested parameters. Samples may be calculated as one or more mixtures, dependent upon the project requirements. Samples must be quantitated against a standard that encompasses the same hydrocarbon range as the range the client has requested. For example, Diesel Range Organics is defined as C10-C28. If a client is interested in the range C12-C14, the DRO standard cannot be used for the quantitation. It is not acceptable to integrate only over the C12-C14 range of the DRO envelope. The narrower range must be quantitated using the specified range in the RT marker standard, as is done in the Oklahoma method. If this approach does not

meet the method requirements of the other methods referenced in this SOP, the client must approve the approach and this approval must be included in the project records.

10.2.1.2 The lowest calibration concentration is equal to the laboratory reporting limit (RL) concentration. The highest standard defines the highest sample extract concentration that may be reported without dilution.

10.2.1.3 It is not acceptable to remove points from a calibration curve for the purpose of meeting criteria, however, the lowest point or the highest point may be removed and the RL or maximum concentration adjusted accordingly (as long as the minimum number of calibration points is sufficient for the calibration model used). Transfer calibration standard solutions into autosampler vials and load onto the GC autosampler. Use the Chem Station software to set up the analytical sequence.

10.2.2 The external standardization method is used. Tabulate the area response for each calibration level against the concentration injected. The ratio of the response to the concentration injected, defined as the calibration factor (CF), is calculated for the standard at each concentration as follows:

$$CF_i = \frac{A_{fuel}}{C_{fuel}} \qquad \textit{Equation 1}$$

Where:

CF_i = Calibration factor for the ith calibration level.
A_{fuel} = Total area of the fuel calibration standard peak.
C_{fuel} = Concentration of fuel calibration standard, mg/mL

10.3 Establishing the Calibration Function

Calibrations are modeled either as average calibration factors or as calibration curves, using a systematic approach to selecting the optimum calibration function. Start with the simplest model, i.e., a straight line through the origin and progress through other options until the calibration criteria are met. Pay particular attention to the residuals noticed at the upper and lower end of the curves. This may be cause for rejection of a curve fit even if the calibration acceptance criteria are met.

10.3.1 Linear Calibration Using Average Calibration Factor

Tabulate the peak area response for each target analyte or hydrocarbon range in each calibration level against the concentration injected. For each analyte in each calibration standard, calculate the calibration factor (CF) as shown in section 10.2.2 above. The calibration factor is a measure of the slope of the calibration line, assuming that the line passes through the origin. Under ideal conditions, the factors calculated for each calibration level will not vary with the concentration of the standard. In practice, some variation can be expected. When the variation, measured as the relative

standard deviation, is relatively small (e.g., ≤ 20%), the use of the straight line through the origin model is generally appropriate.

For each target analyte, calculate the average calibration factor as follows:

$$AverageCalibrationFactor = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n} \qquad \textit{Equation 2}$$

Where:

CF_i = Calibration factor for the ith calibration level.
n = The number of calibration levels.

The relative standard deviation (RSD) is calculated as follows:

$$RSD = \frac{SD}{\overline{CF}} \times 100\% \qquad \textit{Equation 3}$$

Where SD is the standard deviation of the average CF, which is calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(CF_i - \overline{CF}\right)^2}{n-1}} \qquad \textit{Equation 4}$$

10.3.2 If the percent relative standard deviation (%RSD) for the average (mean) of the calculated calibration factors is <u>less</u> than 20%, the average calibration factor can be used for sample quantitation. Methods AK102 and AK103 require the % RSD to be <25%.

10.3.3 Evaluation of the Average Calibration Factor

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average calibration factor. Examine the residuals, i.e., the difference between the actual calibration points and the plotted line. Particular attention should be paid to the residuals for the highest points, and if the residual values are relatively large, a linear regression should be considered. SW-846 Method 8000B allows evaluation of the grand average RSD across all analytes, but TestAmerica-Denver evaluates each analyte individually.

Acceptance Criteria: The RSD must be ≤ 20% (AK102/AK103 limit is ≤ 25%).

Corrective Action: If the RSD exceeds the limit, linearity through the origin cannot be assumed, and a least-squares linear regression should be attempted.

10.3.4 Linear Calibration Using Least-Squares Regression

Calibration using least-squares linear regression produces a straight line that does not necessarily pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression produces the slope and intercept terms for a linear equation in the following form:

$$y = ax + b \qquad \textit{Equation 5}$$

Where:

- y = Instrument response (peak area or height).
- x = Concentration of the target analyte in the calibration standard.
- a = Slope of the line.
- b = The y-intercept of the line.

For an external standard calibration, the above equation takes the following form:

$$A_s = aC_s + b \qquad \textit{Equation 6}$$

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equation, where C_s is now C_e, the concentration of the target analyte in the unknown sample extract.

$$C_e = \frac{A_e - b}{a} \qquad \textit{Equation 7}$$

Where:

- A_s = Area of the chromatographic peak for the target analyte in the calibration standard.
- A_e = Area of the chromatographic peak for the target analyte in the sample extract.
- a = Slope of the line as determined by the least-squares regression.
- C_s = Concentration of the target analyte in the calibration standard.
- C_e = Concentration of the target analyte in the sample extract.
- b = Intercept of the line as determined by the least-squares regression.

10.3.5 Linear Regression Evaluation

With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low

concentrations. Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of a weighted regression over the use of an unweighted regression."

Acceptance Criteria: To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit, and preferably less than the MDL.

Examine the residuals, paying particular attention to the residuals at the low end of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

The linear regression must have a correlation coefficient (r) ≥ 0.990. Some programs (e.g., DoD) require a correlation coefficient ≥ 0.995. DoD QSM 5.0 requires r^2 >0.99. Note that the AK102/AK103 method requires that r^2 be greater than or equal to 0.995.

Corrective Action: If the correlation coefficient falls below the acceptance limit, linear regression cannot be used and a second-order regression should be attempted.

10.3.6 Non-Linear Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$y = ax^2 + bx + c \qquad \textit{Equation 8}$$

Where:

a, b, and c are coefficients determined using a statistical regression technique; y is the instrument response; and x is the concentration of the target analyte in the calibration standard.

10.3.7 Non-Linear Calibration Evaluation

A minimum of six points must be used for a second-order regression fit.

Second-order regressions should be the last option. Note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

Before selecting a second-order regression calibration model, it is important to ensure the following:

- The absolute value of the intercept is not large relative to the lowest concentrations being reported.
- The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).
- The distribution of concentrations is adequate to characterize the curvature.

Acceptance Criteria: The coefficient of determination r^2 must be ≥ 0.990.

Corrective Action: If the coefficient of determination falls below the acceptance limit and the other calibration models are unacceptable, the source of the problem must be investigated and the instrument recalibrated. Third-order regressions are not allowed at TestAmerica Denver.

Note: Method 8015C and the NWTPH method require that the calibration points be "back calculated" to the line or curve and that each point be within +/- 20% (for 8015C) and 15% (for NWTPH) of the expected concentration for that point. See the specific method for how to apply any exceptions (involves a narrowing of the concentration range to within the area that is within control).

10.3.8 Second-Source Initial Calibration Verification (ICV)

10.3.8.1 A second-source initial calibration verification (ICV) standard is prepared as described in section 7.2.16 and analyzed immediately after each ICAL. This standard can also be used as the continuing calibration verification (CCV) standard. The recovered concentration for this standard must be within ± 20% of the expected (or theoretical) concentration (includes DoD QSM 4.2 and 5.0). AK102/AK103 method requires the standard response to be within ± 25% and 8000B requires ± 15%.

10.3.8.2 The percent difference between the measured ICV calibration factor (or the measured concentration of the ICV standard) and the ICAL calibration factor (or the known concentration of the ICV standard) is calculated as follows:

$$\text{Percent Difference} = \frac{R_1 - R_2}{R_1} \times 100\% \qquad \textit{Equation 9}$$

Where:

R_1 = Average calibration factor from the calibration curve or the ICV known value.

R_2 = Calculated calibration factor for the ICV analysis or the measured ICV value.

10.3.8.3 If the percent difference for the second-source verification falls outside of ± 20%, 25% for AK102/AK103 ± 15% for 8000B, then sample analysis cannot be performed. Reanalyze the second-source verification standard to confirm the original result. If the second result fails, then re-prepare the verification standard, and/or re-prepare and rerun the ICAL. The ICV must be analyzed under the same conditions that were used for the ICAL.

10.3.9 Continuing Calibration Verification (CCV)

10.3.9.1 A CCV standard (see Sections 7.2.13 through 7.2.15 for CCV standard concentrations) is analyzed at the beginning of the analytical sequence, every 12 hours of operation, or every 20 samples (whichever is more frequent), and at the end of the analytical sequence. A CCV must be included in each bracket for each fuel type that is requested and quantified for the samples and QC in the bracket.

10.3.9.2 The 8000 series methods (and some state specific methods-Arizona) indicate the need for two levels for the CCV where non-linear calibrations are used and all other methods indicate a single mid-point CCV. The concentration recovered for this standard must be within ± 20% (25% for AK102/AK103) of the expected concentration (see equation 9, above).

Note: It is not necessary to run a CCV standard at the beginning of the sequence if samples are analyzed immediately after the completion of the initial calibration.

10.3.9.3 If the percent difference between the measured CCV value and the expected CCV value falls outside of the method criteria, first check the accuracy of the preparation of the CCV standard. Method criteria for %D for the CCV are:

- ± 20% for Method 8015C or 8015D (Method 8000C), DoD QSM 4.2 or 5.0
- ± 25% for AK102/AK103
- ± 15% for Method 8015B (8000B) and NWTPH

If the standard is accurately prepared and the results fail acceptance criteria then the instrument must be recalibrated and all samples analyzed since the last successful CCV must be

reanalyzed. DoD QSM 5.0 requires recalibration and reanalysis of all affected samples since the last acceptable CCV. As an alternative, the DoD QSM 5.0 indicates that the laboratory may analyze two additional consecutive CCVs within one hour of the failed CCV. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.

If a DoD client accepts TestAmerica's Technical Specifications for DoD QSM work, samples that have no detections when a CCV has recoveries above the project acceptance limits would be reported with a case narrative comment, in addition to applying any data qualifier flags required by the project.

10.3.9.4 In some cases the nature of the samples being analyzed may be the cause of the failing %D. If such matrix effects are suspected then those samples must be reanalyzed (at a dilution if column damage is imminent) to prove matrix effect. If the drift is repeated in the reanalysis, the analyst must generate an NCM for this occurrence to explain that the drift was most likely attributable to the sample matrix and that the samples may be diluted and reanalyzed to minimize the effect if so desired by the client. For DoD QSM 4.2 or 5.1, this must be approved by the client.

10.3.9.5 For any analyte not detected in the client samples, the %D for that analyte in the bracketing CCVs should also be within 20%, however, the results may be acceptable (with client approval) if the drift is positive (high). An NCM must be written to explain this case.

10.4 Retention Time Window Definition

10.4.1 Before establishing RT windows, be certain that the GC system is within optimum operating conditions. Analyze a diesel fuel CCV Standard (Section 7.2.13) and a Retention Time Reference Standard (section 7.2) three times each throughout the course of a 72-hour period. Serial injections over less than a 72-hour period result in retention time windows that are too tight.

10.4.2 Calculate the mean and standard deviation of the three absolute retention times for each compound, carbon range marker, and each surrogate. Table 1 lists the boiling points for the aliphatic hydrocarbons used in the retention time reference standard.

10.4.2.1 The width of the retention time window for an individual peak is defined as ± 3 times the standard deviation of the mean absolute retention time established during the 72-hour period for each component.

10.4.2.2 In those cases where the calculated window for a particular analyte is less than the default window, use ± 0.05 minute (AK102 and AK103) ± 0.03 (8000 methods) and ± 0.1 min (Oklahoma) as the retention time window.

10.4.3 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed, if the instruments conditions are changed significantly, or after a major maintenance event. The data must be retained by the laboratory.

11.0 Procedure

11.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

11.2 Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. An NCM must be used for this documentation.

11.3 Sample Preparation

Refer to extraction SOPs DV-OP-0006 for aqueous samples and DV-OP-0016 and DV-OP-0015 for soil samples. In the case of samples with an organic matrix, waste dilution SOP DV-OP-0012 may apply.

11.4 Instrument Maintenance & Troubleshooting

11.4.1 Before the start of any daily sequence the instrument system should be evaluated for possible maintenance. If the previous run ended with a failing continuing calibration then the system should be maintained to bring it back into control.

- The injector septum should be changed after about 200 injections have been completed.
- If the last CCV that was analyzed indicated a high response then a simple liner change is typically sufficient to bring the system back into control.
- Analysis of a few solvent blanks or a system bake out may be necessary to drive out any residual contamination on the column.
- A reduced response may indicate that the system needs to be evaluated

for leaks.

- Poor peak shape may necessitate clipping a loop out of the analytical column. If this fails to solve the peak shape problem then replacement of the columns may be indicated.

11.4.2 The goal is to maintain the system as close to top condition as possible as was observed when new columns and injector parts were installed. Re-calibration should not be used to correct for maintenance related issues.

11.4.3 Always document any maintenance procedure in the maintenance logbook.

11.5 Analytical Sequence

11.5.1 The analytical sequence starts with a RT reference standard, an initial calibration (ICAL) and initial calibration verification (ICV) or with a continuing calibration verification (CCV) (see Section 10).

11.5.2 An instrument blank (injection of methylene chloride solvent) must be included in each analytical sequence. The instrument blank must meet the same acceptance criteria as the method blank (with the exception of surrogate recoveries).

11.5.3 A CCV standard is interspersed in the analytical sequence after 12 hours have elapsed or after 10 samples have been analyzed, whichever is more frequent. More frequent analysis of the CCV is recommended (and required by some programs) in order to minimize the number of samples needed to be reanalyzed in the event of an out of control recovery for the CCV. A beginning and ending Continuing Calibration Standard must be analyzed for all required carbon ranges.

11.5.4 Any sample suspected of being highly concentrated should be followed by an instrument blank to prevent carryover. If the blank analysis shows contamination beyond established "blank acceptance criteria", the column must be baked out and subsequent blanks analyzed until the system is shown to be free from contaminants. If possible do not aliquot the entire sample volume. Use limited volume autosample vials so that an unused volume remains for possible repeat analyses.

11.5.5 If the measured concentration of any sample exceeds the highest calibration standard concentration, the sample extract must be diluted with methylene chloride and reanalyzed. See section 12.2 for dilution guidelines.

11.5.6 The analytical sequence is closed with a final CCV for each fuel type requested and quantified.

11.6 Daily Retention Time Windows

11.6.1 At the beginning of each daily analytical sequence, the RT Reference Standard ([RTC] Section 7.2) is analyzed. The retention time windows for each diesel / motor oil range are adjusted based on the analysis of each n-alkane. The center of the retention time windows for the jet fuels are adjusted based on the analysis of the chromatography of any level of the jet fuel calibration standard. The center of the retention time windows must be updated at the beginning of each analytical sequence (CCVRT) but not for any other CCV standards.

11.7 Sample Analysis

11.7.1 Baseline Used for Integration

11.7.1.1 The same type of baseline must be applied equally to standards and samples. It is important that the baseline is drawn consistently by each analyst in the GC group, which must be part of the training for new analysts.

11.7.1.2 For the analysis of samples containing hydrocarbons that completely elute in the C_{10} to C_{28} range, the baseline should be drawn as shown in the example in Attachment 1. For routine analysis, this should be similar to the baseline produced by a method blank. Note that because of column bleed, this is not a flat, horizontal common baseline.

11.7.1.3 Measure the area of the methylene chloride blank projecting a horizontal baseline across the retention time range for DRO.

11.7.1.4 Valley-to-valley baselines should not be used. See the example in Attachment 2. Correct the instrument integration settings to minimize the number of manual integrations as shown in the example.

11.7.1.5 Samples containing high concentrations of hydrocarbons heavier than diesel do not completely elute in the C_{10}-C_{28} range. Precise and consistent integration can be difficult. Construction of the baseline requires some experience and judgment on the part of the analyst. Unless otherwise instructed in the client requirements, baseline to baseline integration will be used.

11.7.1.6 When such samples are encountered, it may be necessary to run an additional solvent blank to be sure that there is no carryover between samples.

11.7.1.7 Most of the methods, including SW846 method 8015 require construction of a baseline equivalent to that observed in the method blank (see the example in Attachment 3).

11.7.1.8 Unresolved hydrocarbons should be included in the total concentration. Construction of a common baseline from the signal at the beginning of the pattern to the signal at the end of the pattern (see the example in Attachment 4) should not be performed unless specifically required in the project.

11.7.1.9 Always consult special project instructions for requirements for the baseline.

11.7.2 Confirmation

Second column confirmation is not normally performed for this method as confirmation of chemical identity does not have to be specific.

11.7.3 Data Review

11.7.3.1 First level review is conducted by the analyst to ensure that all acceptance criteria are met, the analysis is properly documented, and the data are correctly uploaded into the LIMS. This is documented using the checklist prescribed in SOP DV-QA-0020, Organic Data Review. The data package should consist of the checklist, sequence(s), ICAL review cover, and any other pertinent communication about the data preparation.

11.7.3.2 Submit the data package for review. Second level review is conducted by the supervisor of the group or an analyst appointed by the supervisor. All manual integrations must be evaluated by the peer reviewer and this review must be documented on the data review checklist. Details of this review and the documentation are described in SOP DV-QA-0020. If problems are found with the data package, then the data are reviewed with the analyst and corrections are made.

11.7.3.3 Other reviews (PM Level III review and QA Data Reviews) are described in the TestAmerica Quality Assurance Manual (QAM), and associated TestAmerica policies and procedures.

12.0 Calculations / Data Reduction

12.1 Calculations

12.1.1 LCS and Surrogate Spike Recovery Calculation

LCS and surrogate spike recoveries are calculated using the following equation:

$$\%\text{Recovery} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) spiked}} \times 100\% \qquad \textit{Equation 13}$$

12.1.2 MS and MSD Recovery Calculation

Matrix spike recoveries are calculated as follows:

$$\text{MS or MSD \%Recovery} = \left(\frac{SSR - SR}{SA}\right) \times 100\% \qquad \textit{Equation 14}$$

Where:

SSR = Measured concentration in spiked sample.

SR = Measured concentration in unspiked sample.

SA = Concentration of spike added to sample.

12.1.3 MS/MSD RPD Calculation

The relative percent difference between the MS and MSD is calculated as follows:

$$\%RPD = \frac{|R_1 - R_2|}{\frac{1}{2}(R_1 + R_2)} \times 100\% \qquad \textit{Equation 15}$$

Where:

R_1 is the result for the MS and R_2 is the result for the MSD.

12.1.4 Concentration of Analyte in the Sample Extract

Depending on the calibration function used, the concentration of the analyte in the sample extract is calculated as follows (see Section 10.3 for details on establishing the calibration function):

Average Calibration Factor: $C_e = \frac{A_s}{CF}$ *Equation 16*

Linear Regression: $C_e = \frac{[A_s - b]}{a}$ *Equation 17*

Non-Linear Regression: $C_e = f(A_s)$ *Equation 18*

Where:

C_e = Concentration of the analyte in the sample extract (ng/mL).

A_s = Peak area for the analyte in the sample extract injection.

b = y-intercept of the calibration fit.

a = Slope of the calibration fit.

$f(A_s)$ = Mathematical function established by the non-linear regression.

12.1.5 Concentration of Analyte in Original Sample (for 1 µL injection)

$$C_{sample} = \frac{C_e}{1000\frac{ng}{\mu g}} \times \frac{V_e}{V_s} \times DF \qquad \textit{Equation 19}$$

Where:

C_{sample} = Concentration of analyte in original sample (µg/L or µg/kg).
C_e = Concentration of analyte in sample extract injected in GC (ng/mL).
$1000\frac{ng}{\mu g}$ = Factor to convert ng/mL to µg/mL.
V_e = Volume of sample extract (mL).
V_s = Volume (or weight) of original sample (L or kg).
DF = Dilution Factor (post extraction dilutions)

12.2 Calibration Range and Sample Dilutions

12.2.1 If the concentration of any analyte exceeds the working range as defined by the calibration standards, then the sample must be diluted with methylene chloride (record the lot number in the run sequence) and reanalyzed. Dilutions should target the most concentrated analyte in the upper half (over 50% of the high level standard) of the calibration range. Samples that were analyzed immediately following the high sample must be evaluated for carryover. If the samples have results at or above the RL for the analyte(s) that were found to be over the calibration range in the high sample, they must be reanalyzed to rule out carryover, unless other objective evidence indicates that the detection is not the result of carryover. Such evidence may include an observation where carryover was not observed when samples or blanks were analyzed after another sample with similar high compound recovery or when the detection in the sample with suspected carryover is much higher than the expected amount of carryover (i.e., the sample's concentration may be similar to or higher than the concentration found in the previous sample). It may also be necessary to dilute samples because of matrix interferences.

12.2.2 If the initial diluted run has no hits or hits below 20% of the calibration range, and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

13.0 Method Performance

13.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.2 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.2.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.2.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.2.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.2.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.2.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.2.6 The Oklahoma method requires that the analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

13.2.6.1 Analysis of 7 replicates of the diesel component standard at a concentration of 100 ug/L of total DRO in organic-free water with recoveries of all components within ± 40% of the known concentration and precision of all replicates within ± 30%.

13.2.6.2 Analysis of 7 replicates of DRO_free sand at a concentration of 10 mg/kg of total DRO with all recoveries within ± 40% of the known concentration and precision of all replicates within ± 30%.

13.2.6.3 As an exception use the NELAC recommended procedure for initial demonstration of capability in place of this procedure.

13.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Environmental Health and Safety Manual (M-E-001 DV) for "Waste Management and Pollution Prevention."

14.2 Standards and reagents are prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Vial waste – Expired Extract Vials – Waste Stream A

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 Determinative Chromatographic Separations, , SW-846, Test Methods for

Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.1.1 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996

16.1.2 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.1.3 Method 8015B, Nonhalogenated organics by Gas Chromatography, Revision 2, December 1996.

16.1.4 Method 8015C, Nonhalogenated organics by Gas Chromatography, Revision 3, February 2007.

16.1.5 Method 8015D, Nonhalogenated organics by Gas Chromatography, Revision 4, June 2003.

16.1.6 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.1.7 Method 3546, Microwave Extraction, Revision 0, February 2007.

16.1.8 Method 3550B, Ultrasonic Extraction, Revision 3, December 1996.

16.1.9 Method 3550C, Ultrasonic Extraction, Revision 4, February 2007.

16.2 Alaska Method AK102, "For the Determination of Diesel Range Organics", Version 04/08/02.

16.3 Alaska Method AK103, "For the Determination of Residual Range Organics", Version 04/08/02.

16.4 NWTPH-HCID "Hydrocarbon Identification Method for Soil and Water," Manchester Environmental Laboratory, Dept. of Ecology, State of Washington.

16.5 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified), Diesel Range Organics (DRO), October 22, 1997, Rev. 4.1.

16.6 Kansas Department of Health and Environment, "Kansas Method for the Determination of Mid-Range Hydrocarbons (MRH) and High-Range Hydrocarbons (HRH)" Revision 1.0 Nov 2015

17.0 Modifications from Source Methods

17.1 Alaska Method AK103 for residual range organics (RRO) does not include analysis of aqueous samples. This SOP does provide for the determination of RRO, as defined in the Alaska method, in water.

17.2 The NELAC IDOC procedure is used in place of the Oklahoma IDOC procedure.

17.3 Method AK102 calls for the LCS to be the same source as used for the calibration stock. The laboratory typically uses second source standards for the LCS.

17.4 Method AK102 requires standards to be stored in the freezer. The laboratory stores all standards in the refrigerator as recommended by the manufacturer and other source methods. The same stock standards are used for all analyses.

18.0 Attachments

Table I:	Aliphatic Hydrocarbon Standard
Table II:	Calibration Levels
Table III:	Spike Levels for Quality Control
Table IV:	Recommended GC Conditions
Table V:	Alaska Methods AK102 and AK103 Acceptance Criteria for Quality Control
Table VI:	DRO and RRO Method Summary Comparison Chart
Table VII:	DoD QSM 5.0 Requirements for Organic Analysis by Gas Chromatography
Attachment 1:	Routine DRO Integration
Attachment 2:	Valley-to-Valley Integration Cannot Be Used
Attachment 3:	Heavy Hydrocarbons with Baseline from Method Blank
Attachment 4:	Heavy Hydrocarbons with Non-Standard Signal-to-Signal Integration
Attachment 5:	Retention Time Reference Standard
Appendix 1:	Oklahoma Method Specifications

19.0 Revision History

- Revision 10, dated 3/3/2020
  - Removed all Kansas MRH/HRH information and tables. Method has been deactivated. References and Revision History for Kansas MRH/HRH were not removed.
  - Update column information for instrument U in section 6.2.1.2
  - Sections with DOD QSM 5.0 were updated to the most recent QSM 5.1
  - Section 11.5.3 had a 20 sample bracket listed. Changed to 10 sample injection per sequence bracket.
  - Section 11.6.2 was removed as it was already included in 11.6.1
  - Removed Bunker C information from section 3.5. Denver does not analyze Bunker C.
  - Updated names for approval signatures
  - Updated copyright information
  - Annual Review
- Revision 9, dated 2/28/2019
  - Revised section 7.2 in order to report the concentration of standards for narrower Carbon ranges as a percentage of the concentration of the widest range for DRO and RRO
  - Annual Review
- Revision 8, dated 2 May 2018

- Annual Review.

- Revision 7, dated 31 March 2017
  - Added requirements for KS MRH/HRH throughout.
  - Updated column descriptions in Section 6.2.1
  - Added Hexane to Reagents in Section 7.1.3 for KS method
  - Updated Matrix Spike/Matrix Spike duplicate corrective actions in Section 9.3.4 to reflect with current policy

- Revision 6, dated 31 December 2015
  - Added detail to Section 2.1 regarding sample sizes
  - Added detail to the definition of JP-8 (Section 3.4)
  - Added SOP reference for Silica Gel cleanup if performed to section 4.1
  - Removed reference to JP4 in Section 7.
  - Added details to Section 7.2.14 for second source calibration verification standards
  - Added section 7.5.6
  - Corrected sample size for solid matrix in section 9.3.3.2
  - Added requirement that ICAL be performed at least annually per the QA Manual (Section 10.1.7)
  - Clarified language in Section 10.3.9.3 regarding corrective action for CCV failures
  - Deleted Section 11.7.2 and renumbered remainder of Section 11. The information on calculations was duplicated in Section 12.
  - Added Method modifications for AK102 (Sections 17.3 and 17.4)
  - Removed reference to TX1005 from Table VI. Laboratory no longer performs this method.
  - Added Table VII, summarizing QSM requirements and standard variances (if accepted by the client) and specific to this procedure

- Revision 5, dated 31 December 2014
  - Removed references to CA LUFT method. Lab does not perform.
  - Removed information and procedure steps related to analysis of Terphenyls. Lab no longer performs.
  - Removed client specific references to a separate work instruction.
  - Updated instrumentation descriptions and supplies list.
  - Revised section 7.3 to reflect current practice
  - Reformatted Section 8.0 as a table for ease of reference
  - Added details regarding requirements for DoD QSM 4.2 and 5.0 throughout
  - Added "carbon range marker" to Section 10.4.2 when calculating retention time windows
  - Added requirement the RT windows must be recalculated when column replaced, significant change to instruments or after major maintenance.
  - Removed requirement for L2 reviewer to sign and date the MI summary report. Review is documented on the data review checklist.

- Revision 4, dated 31 October 2013
  - Editorial and formatting updates
  - Updates throughout the document to reflect current practices

- Revision 3.2, dated 15 October 2012
  - Updated section 1.2 to include analysis up to C_{40}.
  - Updated section 3.0. Updated the ranges for DRO, deleted the option for JP4, added the ranges for Bunker C.
  - Updated section 11.6.1.7 to clarify appropriate baseline assignment.
  - Updated section 11.6.1.8 to clarify the requirement to include unresolved hydrocarbons in the integration.
  - Change the name of Appendix 4 to "Heavy Hydrocarbons with Non-Standard Signal-to-Signal Integration"
- Revision 3.1, dated 30 June 2012
  - Updated to include Terphenyls
- Revision 3, dated 04 April 2012
  - Updated section 6.2 with current supply list.
  - Added some current vendor sources in section 7.2 for materials used.
  - Added sections 7.3 Standards Verification, 7.4 Storage of Stock Standards, and section 7.5 Non-Routine Compounds.
  - Added holding time clarification to section 8.3.
  - Modified Section 10 for clarity and consistency with other SOP formats. Removed wording from section 10.3.4 that indicated the need for at least three multi-point curves in order to use a weighted regression. Changed the wording for section 10.4.2.2 to indicate when to use a default RT window.
  - Modified section 11 to include some guidelines for 11.3 Instrument Maintenance.
  - Modified section 12 to be consistent with other SOP formats.
  - Updated Table IV with current configurations.
  - Added Table VI and Attachment 5
- Revision 2, dated 25 March 2011
  - Updated SOP to include requirements for the various revisions of Methods 8000, 8015 and to include state methods for extractable hydrocarbons, including Alaska, California, Oklahoma and Washington.
  - Modified definitions for clarity
  - Updated section 13.
- Revision 1, dated 05 March 2010
  - Added section 6.3
  - Updated to match other GC SOP Format
- Revision 0, dated 31 January 2009

Table I: Aliphatic Hydrocarbon Standard

Compound Boiling Points		
n-Alkane	**Name**	**BP_{760} (°C)**
C_8	Octane	126
C_{10}	Decane	174
C_{24}	Tetracosane	391
C_{28}	Octacosane	432
C_{32}	Dotriacontane	468
C_{36}	Hextriacontane	498

This Table can be used to get the estimated boiling point ranges of the hydrocarbons reported in a given sample.

Table II: Calibration Levels

Standard	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
DRO (in mg/mL)	0.1	0.25	1.00	5.00	7.50	15.00	30.00
Jet Fuel 8 (in mg/mL)	0.1	0.5	1.00	2.50	5.00	10.00	NA
Motor Oil / RRO (in mg/mL)	0.1	0.25	1.00	5.00	7.50	15.00	30.0
Ortho-Terphenyl and n-octacosane (surrogates) (in mg/mL)	0.0005	0.00125	0.005	0.025	0.0375	0.075	---
KS MRH/HRH (in µg/L)							
MRH	30	60	150	300	630	900	1200
HRH	45	90	225	450	945	1350	1800
1-Chlorooctadecane (surrogate)	5	10	20	40	105	150	---

Table III: Spike Levels for Quality Control

Laboratory Control Samples (LCS) and Matrix Spike/ Spike Duplicate (MS/MSD)		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Soil (mg/kg)**
Diesel Range Organics	2.0	66.7
Jet Fuel 8	1.0	33.3
Residual Range Organics (or Motor Oil)	5.0	166.5

Surrogate Analytes		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Low Soil (mg/kg)**
ortho-Terphenyl	0.02	0.667
n-Octacosane	0.02	0.667
1-Chlorooctadecane	0.04	1.33

Table IV: Recommended GC Conditions

Component	Condition	Instrument
Hydrogen Column Pressure	20 psi	U2
	15 psi	U
Initial Column Temperature	45 ºC for 2.5 minutes	U2
	45 ºC for 2 minutes	U
Temperature Ramp	Ramp 25 ºC / minute to 325 ºC, hold for 3.3 minutes	U2
	Ramp 35 ºC/minute to 300 ºC, hold 4 minutes, ramp 35 ºC to 330 ºC, hold 4.5 minutes	U
Final Column Temperature	325 ºC for 3.3 minutes	U2
	330 ºC for 4.5 minutes	U
Injector Temperature	285 ºC	U2
	280 ºC	U
FID Temperature	325 ºC	U2 & U

Table V. Alaska Methods AK102 and AK103 Acceptance Criteria for Quality Control

Method AK102 (DRO) Acceptance Criteria				
QC Parameter	**Water (mg/L)[1]**	**Soils (mg/kg)[1]**	**% Recovery**	**RPD (%)**
Lab Fortified Blanks (LCS)	0.5 – 2.0	---	75 - 125	20
Continuing Calibration (Includes surrogate compounds)[2]	---	---	75 - 125	---
Calibration Verification	---	---	75 - 125	---
Surrogate Recovery for "Laboratory Control Sample"[2]	0.02	0.8	60 - 120	---
Surrogate Recovery for Field Samples	0.02	0.8	50 - 150	---
Method AK103 (RRO) Acceptance Criteria				
QC Parameter	**Water (mg/L)[1]**	**Soils (mg/kg)[1]**	**% Recovery**	**RPD (%)**
Lab Fortified Blanks (LCS)	---	500	60 - 120	20
Continuing Calibration	2000	---	75 - 125	---
Calibration Verification	2000	---	75 - 125	---
Surrogate (n-triacontane-d_{62}) Recovery for Control Samples[2]	---	50	60 - 120	---
Surrogate (n-triacontane-d_{62}) Recovery for Field Samples	---	50	50 - 150	---

NOTE: The information in this table is taken verbatim from the referenced Alaska methods. This SOP provides for spiking both DRO and RRO in water and soils. The acceptance limits in this table are used when client or project requirements specify compliance with the Alaska methods.

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[1] Suggested concentrations. May vary with matrix.

[2] According to the Alaska methods, this is any laboratory prepared samples used for quality control, except calibration standards. At TestAmerica Denver, this includes the LCS, method blank, MS, and MSD. The continuing calibration for surrogates is verified at the instrument by including them in the continuing calibration DRO sample.

Table VI. DRO and RRO Method Summary Comparison Chart

Method	Standards Surrogates	Ranges Min RT Windows	Batch Size	MB RL	ICAL # points, % RSD	CCV	LCS/MS/Other Sample Size
8015B	Commercial Diesel	C_{10}-C_{28} Apex ± window	20 samples	Blank subtraction is allowed	Minimum 5 levels; lowest at RL %RSD ≤ 20	CCV/12-hour window & close; Midrange ± 15% All compounds in RT window	Standard QC model
8015C	Commercial #2 Diesel 6 month expiration	C_{10}-C_{28} Apex ± window	20 samples	Blank subtraction is allowed	Minimum 5 levels; lowest at RL %RSD ≤ 20	CCV/12-hour window & close; Midrange ± 20% All compounds in RT window	Standard QC model
8015D	Commercial #2 Diesel 6 month expiration	C_{10}-C_{28} Apex ± window	20 samples	Blank subtraction is allowed	Minimum 5 levels; lowest at RL %RSD ≤ 20	CCV/12-hour window & close; Midrange ± 20% All compounds in RT window	Standard QC model
8000B	NA	Default 0.03 min; ± 3 standard deviations over 72 hour period. Use center of CCV in beginning or mid-point standard of ICAL *New RT with new column installation; at least annually	20 samples	Less than RL Less than 5% of sample result	Minimum 5 levels; lowest ≤ RL %RSD ≤ 20 (mean RSD allowed) r ≥ 0.99 6 level for quadratic COD ≥ 0.99	CCV/12-hour window & close; Midrange; every 10 samples recommended ± 15% (grand mean allowed) 2 levels for quadratic curve	In-house limits
8000C	NA	Default 0.03 min; ± 3 standard deviations over 72 hour period. Use center of CCV in beginning or mid-point standard of ICAL *New RT with new column installation; at least annually	20 samples	Less than RL	Minimum 5 levels; lowest ≤ RL %RSD ≤ 20; r^2 ≥ 0.99 6 level for quadratic COD ≥ 0.99 Batch calculation is suggested (20% D)	± 20% 2 levels for quadratic curve	In-house limits
DRO/AK102	Commercial #2 Diesel o-terphenyl	Beginning C_{10} to beginning C_{35} ± 0.05 min	20 samples	20 mg/Kg 800 µg/mL Less than PQL	r^2 ≥ 0.995 At least three standards %RSD ≤ 25	ICV ± 25% CCV/CCS ± 25% Every 20 samples Midpoint of the curve	LFB = 60-120%
RRO/AK103	Equal Weights of 30W and 40W motor oil Surrogate: n-Triacontane	Beginning C_{10} to beginning C_{36} ± 0.05 min	20 samples	100 mg/Kg	r^2 ≥ 0.995 At least three standards %RSD ≤ 25	ICV ± 25% CCV/CCS ± 25% Every 20 samples Midpoint of the curve	RL ~ 5x MDL LFB = 60-120%
Oklahoma	Ten component blend of alkanes; 10-28 even (store in freezer) 6 month expiration	0.1 min before C_{10} to 0.1 min after C_{28} (0.1 min)	20 samples	0.1 mg/L 10 mg/Kg Less than PQL	Minimum 3 levels Lowest at PQL r at least 0.99	CCV ± 20% daily Has special IDOC (see method)	Sample size discretionary; at least 800 mL. Soil not to exceed 20 g 1 mL FV LCS/LCSD & MS/MSD ± 20% for water; ± 40% for soil
NWTPH	#2 Diesel or known other component Surrogate: o-terphenyl	Must incorporate most of components; break where multicomponents occur	MB per 20 samples; Duplicate per 10 samples	Diesel: 25 mg/Kg; 0.25 mg/L Motor Oil: 100 mg/Kg; 0.5 mg/L	Minimum 5 points r>0.990 Back quantitate standards to ± 15%	CCV ± 15% Opening and closing Midpoint of the curve	20 g soil 400 mL water
KS MRH	7 component mixture from C_7-C_{16}	0.1 minute before n-nonane to 0.1 minutes before n-nonadecane	20 samples	0.2 mg/L; 1 mg/Kg Less than RL	Minimum 5 points %RSD ≤ 25% Linear regression may be used: Lab required r at least 0.99	CCV: ± 25% Opening and closing	LCS (LCSD) 40-140%, RPD ≤ 25% MS/MSD40-140%; RPD ≤ 50% 1 L (aq); 10 g (s)
KS HRH	8 component mixture from C_{16}-C_{36}	0.1 minutes before n-nonadecane to 0.1minutes after n-pentatriacontane		0.075 mg/L; 5 mg/Kg Less than RL			

Table VII
DoD QSM 5.0 Requirements for Organic Analysis by Gas Chromatography

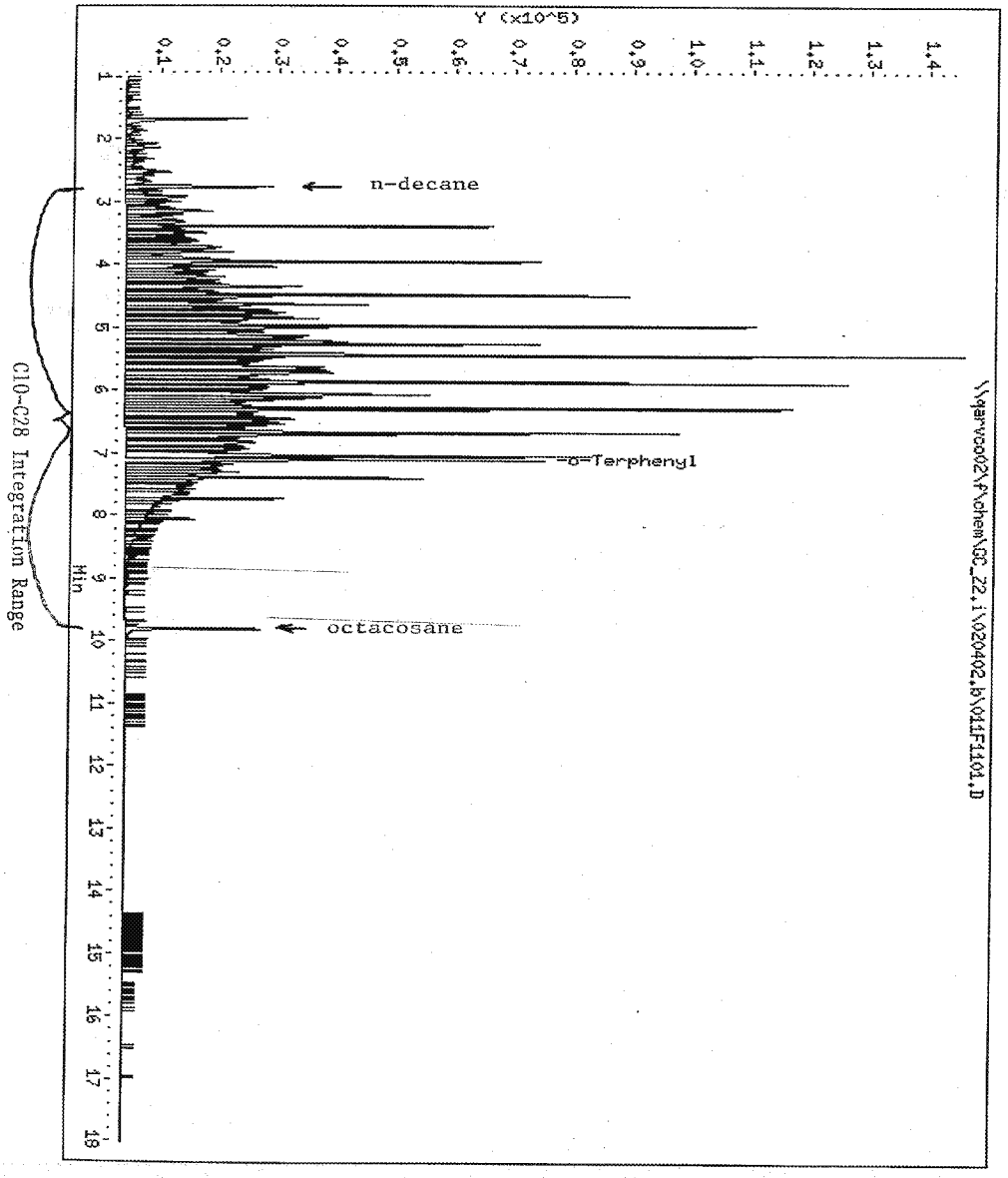
(Includes both QSM requirements and TestAmerica Technical Specifications applicable to this SOP that may be implemented with client approval)

Category	Requirement 8015-DRO and Motor Oil and State Methods
Initial Calibration (ICAL) for all analytes including surrogates	Perform a minimum 5 point calibration for all analytes prior to sample analysis for linear and 6 levels for quadratic at instrument set-up and after ICV or CCV failure, prior to sample analysis. Acceptance Criteria options: 1. RSD for each analyte ≤ 20%. 2. Linear least squares regression: $r^2 \geq 0.99$ (r>0.995) 3. Non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$. This option is not used for Method 8082 or Method 8082A. Any problems must be corrected and ICAL repeated. No samples shall be analyzed until an ICAL has passed.
RT Window Position Establishment	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical sequence. Set position using the midpoint standard of the calibration curve when an ICAL is performed. On days when ICAL is not performed the initial CCV is used.
Retention Time (RT) Window Width	Perform 72-hour study at method set-up and after major maintenance (e.g., column change) to calculate the RT window width for each analyte and surrogate. RT width is ± 3 times the standard deviation for each analyte RT from the 72 hour study.
Initial Calibration Verification (ICV)	Measure a second-source standard once after each ICAL prior to sample analysis. Acceptance limits for all reported analytes is ± 20% of expected value. All reported analytes must be within established RT window. Correct any problems and rerun second-source standard. If that fails, correct problem and repeat ICAL. No samples shall be analyzed until the second-source calibration verification is successful.
Continuing Calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run at beginning of day, after every 10 field samples, and at the end of the analysis sequence. All reported analytes must be within ± 20% of expected value (calculated from ICAL). All analytes must be within established RT windows. If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project. (3HR) Recalibrate and reanalyze all affected samples since the last acceptable CCV; Or Immediately (within one hour) analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. (Passing criteria only has to apply to the analytes that failed in the bracketing CCV.) If either reanalyzed CCV fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV If a CCV fails to meet control limits, the routine corrective action process is to recalibrate and reanalyze all affected samples since the last acceptable CCV. TestAmerica will perform a single reanalysis. If it is determined through reanalysis that project samples are the cause of CCV failures, the client will be consulted before reporting (6CCVOrg) If reanalysis is not possible, apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable CCV. Must be explained in the case narratives.

Table VII
DoD QSM 5.0 Requirements for Organic Analysis by Gas Chromatography (continued)

Category	Requirement 8015-DRO and Motor Oil and State Methods
Method Blank (MB)	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. For Method RSK-175, no common lab contaminants >LOQ (2CLC) If criteria is not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narratives. Flagging is only appropriate when samples cannot be reanalyzed.
LCS	One LCS per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates. For Method 8081, only single-component compounds are spiked (9Spik) Use DoD acceptance criteria if project limits are not specified. If analyte not listed by DoD, use in-house LCS limits if project limits not specified. If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Otherwise, correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Flagging is only appropriate when samples cannot be reanalyzed unless 3HR is accepted by the client.
Matrix Spike	One per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. The MS is for matrix evaluation only. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error.
MSD or Sample Duplicate	One per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates. Use DoD-specific criteria for LCS. For failures, consult project-specific DQOs and contact client for additional measures to be taken. RPD between duplicates ≤ 30%. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. Explain in the case narrative. Data shall be evaluated to determine the source of the difference.
Surrogate Spike	Spike all field and QC samples with surrogates. Must meet DoD acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. If surrogate recoveries are above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project. For samples with ND results, a high bias as evidenced in these situations is typically not an issue (3HR). Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the case narrative.

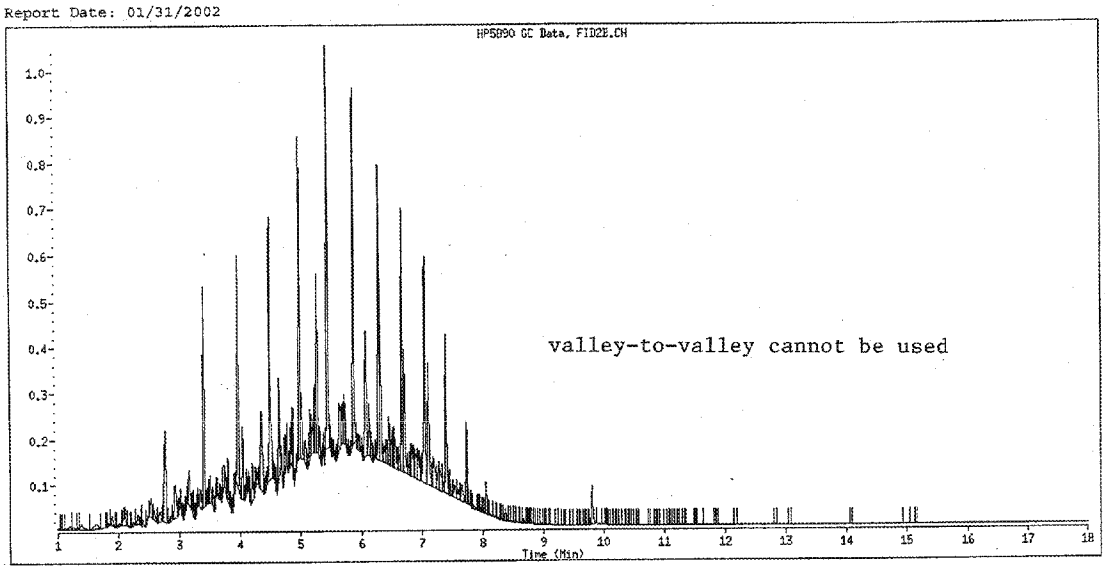
Attachment 1
Routine DRO Integration



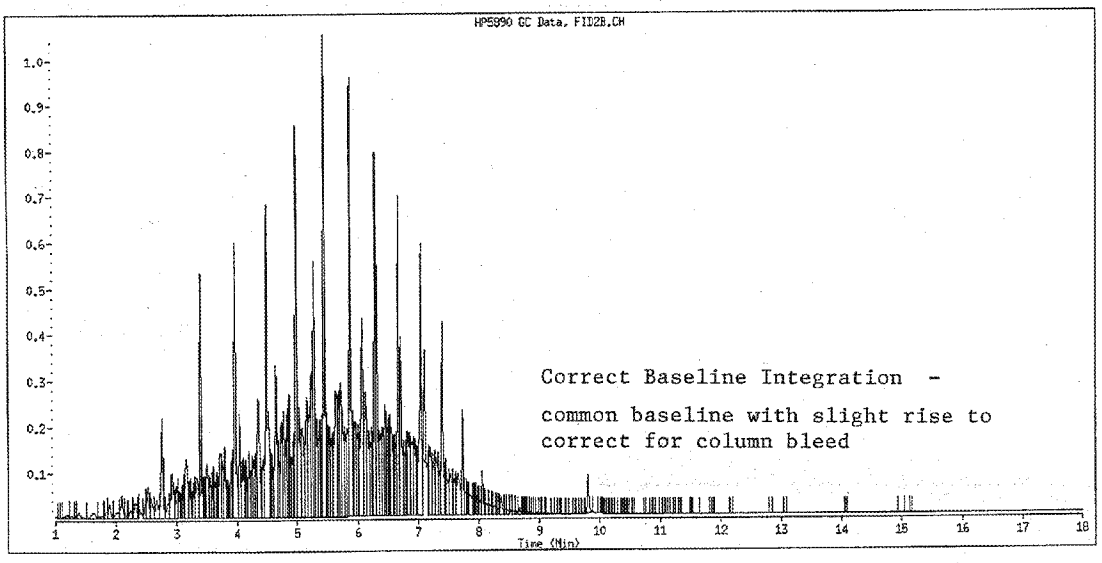
Attachment 2
Valley to Valley Integration cannot be used

MANUAL INTEGRATION TO PRODUCE CORRECT BASELINE

Report Date: 01/31/2002



Original Integration

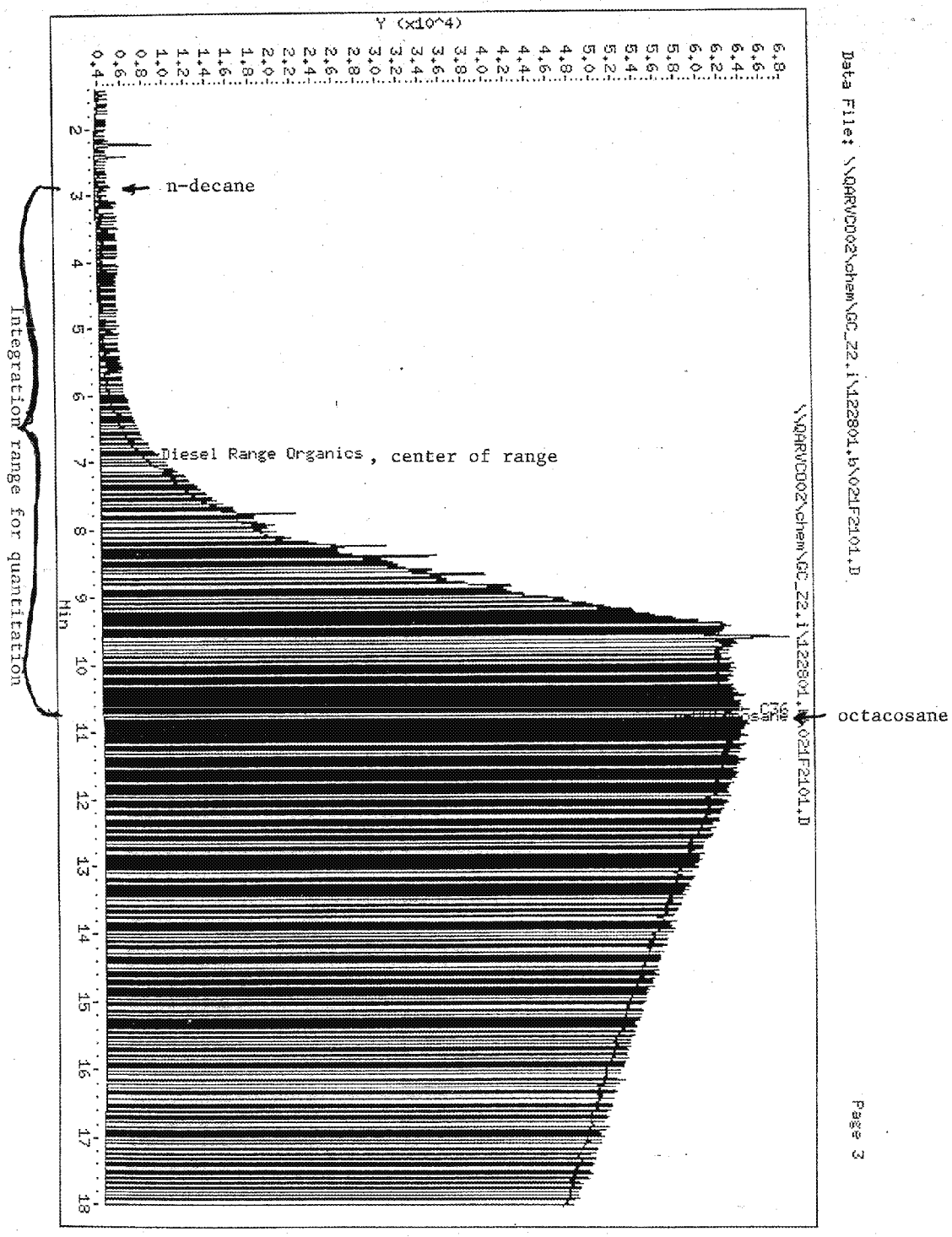


Manual Integration

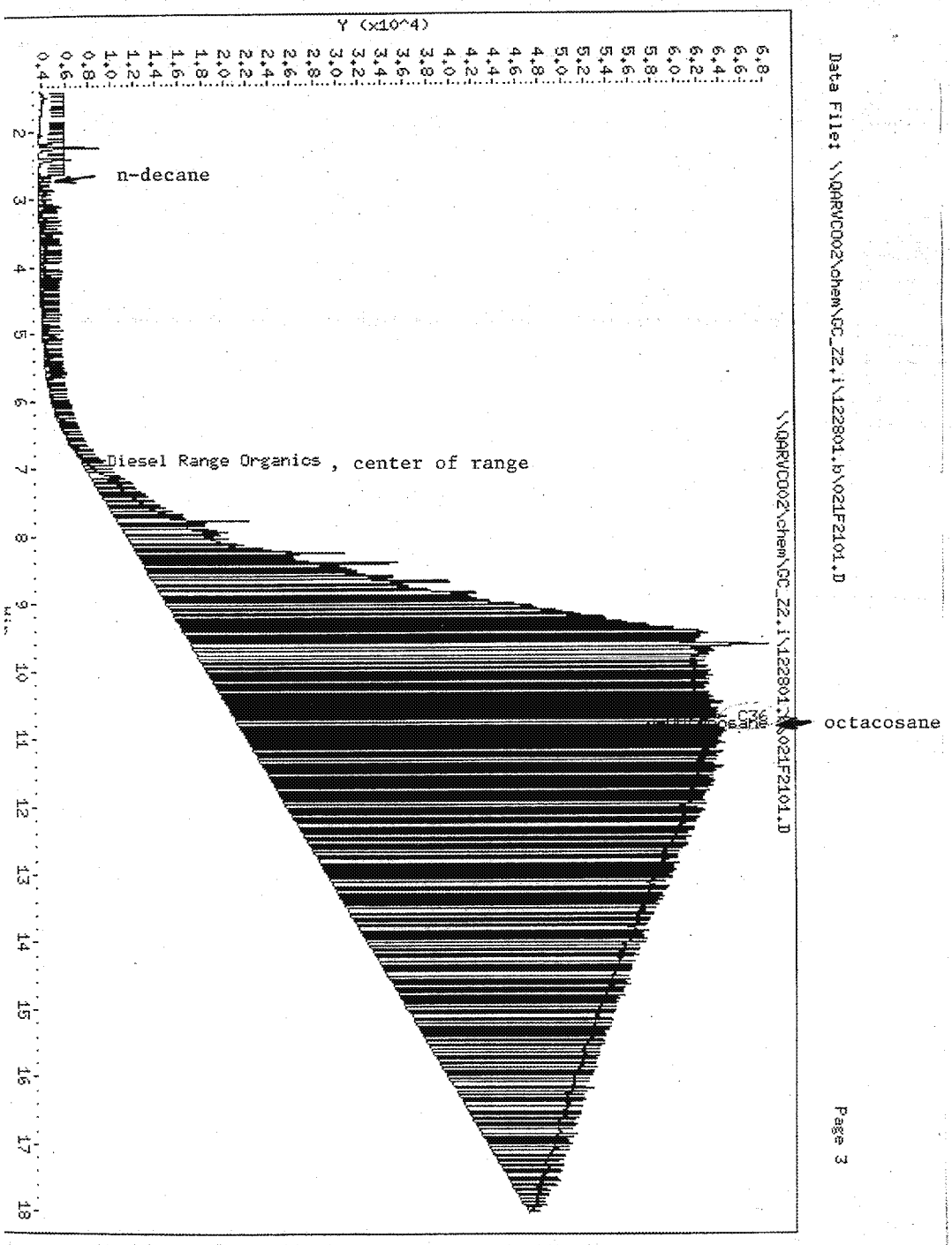
Manually Integrated By: fiedlerh
Manual Integration Reason: Baseline Event

HF 1/31/02

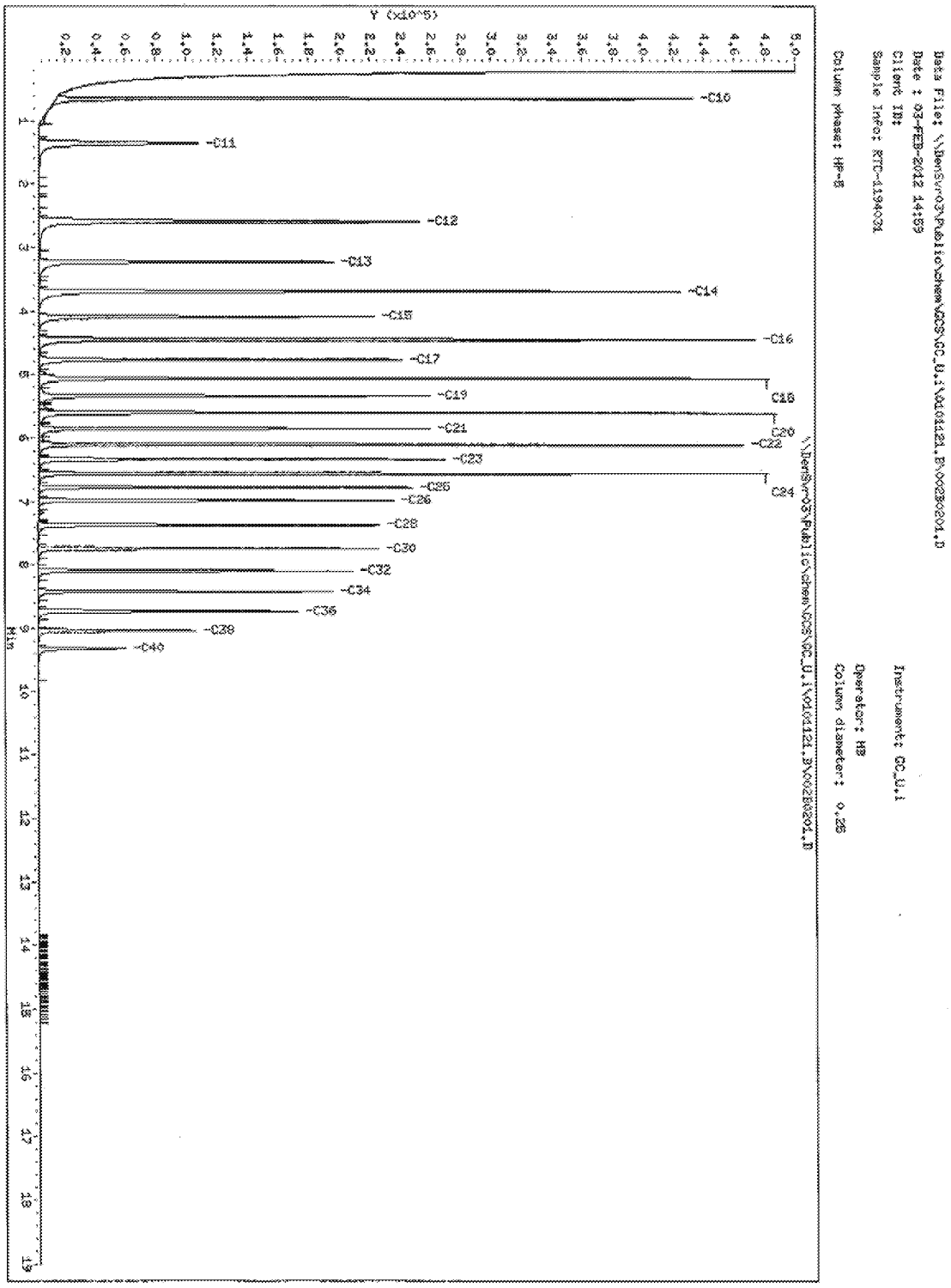
Attachment 3
Heavy Hydrocarbons with Baseline from Method Blank



Attachment 4
Heavy Hydrocarbons with Non-Standard Signal to Signal Integration



Attachment 5
Retention Time Reference Standard



Appendix 1: Oklahoma DRO/RRO Requirements

Definitions

C_8-C_{11}; C_{12}-C_{14}; C_{15}-C_{20}; C_{21}-C_{30}. Each of these carbon ranges are determined by the retention time defining standard. In terms of retention time, the lab will include instrument response eluting 0.1 minutes prior to the apex of the peak for the first alkane of a given carbon range. The end of a given carbon range is defined by the retention time for the alkane marker that designates the beginning of the next carbon range, finally ending 0.1 minutes after the elution of the C_{30} marker. Identification and quantification of TPH components requires more analytical judgment than other GC methods. The TPH chromatograms consist of groups of peaks that fall within a noted carbon retention time range. Diesel Fuel will be defined as C_8-C_{30} for the Oklahoma method. **Please note only range C10-28 is reported for OKLA_DRO.**

The standard used for this calibration is a mixture of the even-numbered n-alkanes from C_{10} – C_{28}. To calibrate for this method, the following determinations are used:

C_8 – C_{11} range: based on the response of C_{10}
C_{12} – C_{14} range: based on the responses of C_{12} and C_{14}
C_{15} – C_{20} range: based on the responses of C_{16}, C_{18} and C_{20}
C_{21} – C_{30} range: based on the responses of C_{22}, C_{24}, C_{26}, and C_{28}

Standards:

Stock Oklahoma Standard: Supelco TPH1 Standard at 2000 µg/mL of each n-alkane compound.

Stock Oklahoma Second Source Standard: Ultra Scientific EPA/Wisconsin Standard at 2000 µg/mL of each n-alkane compound.

Stock Oklahoma Surrogate Standard: Supelco o-terphenyl solution at 10,000 µg/mL in methylene chloride.

Oklahoma Surrogate Spike Solution: Dilute 5 mL of Stock Oklahoma Surrogate Solution to a final volume of 200 mL using 90:10 acetone:methylene chloride for a final concentration of 50 µg/mL.

Oklahoma Spike Solution: Dilute 12.5 mL of Stock Diesel Fuel Second Source Standard to a final volume of 50 mL using acetone for a final concentration of 500 µg/mL of each n-alkane.

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Appendix 1: Oklahoma DRO/RRO Requirements (continued)

Oklahoma Calibration Standards: Diesel fuel standards for the Oklahoma methods are prepared in methylene chloride at 6 concentration levels using the Stock Oklahoma Standard and the Stock Oklahoma Surrogate Standard as defined for this method.

Level	Fuel Standard Solution Used	Volume Used (mL)	Final Volume (mL)	Final Conc of each n-alkane (µg/mL)	Surrogate Conc (µg/mL)
6	Stock + Stock Surrogate	0.75 .15	1.0	1500	1500
5	Level 6	.2	0.6	500	500
4*	Stock + Stock Surrogate	0.25 0.05	5	100	100
3	Level 4	0.5	1.0	50	50
2	Level 4	0.25	1.0	25	25
1	Level 4	0.1	1.0	10	10

NOTE: The Level 4 (*) calibration standard is also used as the continuing calibration verification standard.

Oklahoma Second Source: The second source for this method is prepared by diluting 0.05 mL of the Stock Oklahoma Second Source Standard and 0.01 mL of the Stock Oklahoma Surrogate Standard to a final volume of 1 mL.

Calibration Levels:

Standard	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
$C_8 - C_{11}$	10	25	50	100	500	1500
$C_{12} - C_{14}$	20	50	100	200	1000	3000
$C_{15} - C_{20}$	30	75	150	300	1500	4500
$C_{21} - C_{30}$	40	100	200	400	2000	6000
$C_8 - C_{30}$	100	250	500	1000	5000	15000
Ortho-Terphenyl (surrogate)	10	25	50	100	500	1500

(*All concentrations are in µg/L.*)

Please note only range C10-28 is reported for OKLA_DRO. Please refer to 8015 methods for additional ranges.

Appendix 1: Oklahoma DRO/RRO Requirements (continued)

Spike Levels for Quality Control

Laboratory Control Samples (LCS) and Matrix Spike/ Spike Duplicate		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Soil (mg/kg)**
$C_8 – C_{11}$	0.5	10
$C_{12} – C_{14}$	1.0	20
$C_{15} – C_{20}$	1.5	30
$C_{21} – C_{30}$	2.0	40
$C_8 – C_{30}$	5.0	100

Surrogate Control Samples		
Analyte	**Spike Concentration**	
	Water (mg/L)	**Low Soil (mg/kg)**
Ortho-Terphenyl	0.05	1

Please note only range C10-28 is reported for OKLA_DRO. Please refer to 8015 methods for additional ranges.

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Title: **Determination of Volatile Organics by GC/MS [8260B/C/D and 624/624.1]**

Approvals (Signature/Date):

3/13/20	3/13/20
Gabe Ornelas Technical Manager Date	Reed Pottruff Health & Safety Manager / Coordinator Date
Signing for: 3/13/20	3/13/20
Roxanne Sullivan Quality Assurance Manager Thomas Lill Date	Scott Hall Laboratory Director Date

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1.0 Scope and Application

1.1 This method is applicable to the determination of volatile organic compounds (VOCs) in water, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Table 1. Additional analytes that can be determined by this SOP are listed in Tables 2, 3 and 4.

1.2 This SOP is applicable to Method 8260B/C/D, which is appropriate for compliance testing under RCRA regulations and Method 624/624.1 (CWA compliance testing). It is important that the procedural differences described in this document for these methods are carefully observed.

1.3 This method can be used to quantify most volatile organic compounds that have boiling points below 200 °C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4 The method is based upon a purge-and-trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 0.5 to 200 µg/L for 8260B/C/D waters, 1.0 to 200 µg/kg for low-level soils, and 200 to 30,000 µg/kg for medium-level soils. The working range for Method 624/624.1 (5 mL purge) is 1.0-200 µg/L.

1.5 Reporting limits for Method 8260B/C/D are listed in Tables 1, 2, and 3. Reporting limits for Method 624/624.1 are given in Table A1. Reporting limits for soil samples prepared by the AK methanol technique are listed in Table Bp-1.

1.6 Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates (MS/MSD), and laboratory control spike samples (LCS).

2.0 Summary of Method

2.1 Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the gas chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.2 Aqueous samples are purged directly. Generally, soils are preserved by extracting the volatile analytes into methanol. If especially low detection limits are required, soil samples may be preserved in water (with or without sodium bisulfate) and purged directly.

2.3 In the purge-and-trap process, an inert gas is bubbled through the solution at ambient temperature or at 40 °C (40 °C is required for low-level soils), and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

2.4 Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each

identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

2.5 Unless otherwise noted all requirements are for both 8260B, 8260C, 8260D and 624/624.1.1

3.0 Definitions

3.1 **Contaminated glassware:** Glassware that may have come into contact with samples and/or chemical standard and that have not been cleaned using this procedure.

3.2 **Uncontaminated glassware:** Glassware that has not come into contact with samples, chemical standards, or is otherwise believed to be uncontaminated.

NOTE: Newly purchased glassware is not necessarily clean. The manufacturing process can contaminate glassware with high molecular weight organic constituents that can be extracted by organic solvents. New glassware should be thoroughly Methanol rinsed prior to use for analytical applications.

3.3 Calibration Check Compound (CCC)

CCCs are a representative group of compounds that are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and percent drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.4 System Performance Check Compounds (SPCC)

SPCCs are compounds that are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

3.5 Initial Calibration Verification (ICV)

The ICV is a second-source calibration verification standard. In this SOP, the LCS and the MS/MSD spikes are second-source standards.

3.6 Continuing Calibration Verification (CCV)

A solution of method analytes, surrogate compounds, and internal standards used to evaluate the performance of the instrument system with respect to a defined set of method criteria.

3.7 Lower Limit of Quantitation (LLOQ)

The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be $\geq$ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

3.8 Other Quality Terms

The quality control terms used in this procedure are consistent with SW-846 terminology. Definitions are provided in the glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and in SOP DV-QA-003P, Quality Assurance Program.

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge-and-trap-grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2 It is imperative that all glassware used in the laboratory be free of contaminants and potential interferences before starting and analysis or extraction process. Many problems encountered in the laboratory can be traced to improperly cleaned glassware and can be avoided by following the proper cleaning procedures.

4.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.4 Matrix interferences may be caused by non-target contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.5 Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.6 Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample. (See Section 10.7.4.12.)

4.7 Interferences are observed with the surrogate Toluene-d_8 when the samples appear to be treated with potassium permanganate.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.2 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.3 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.3.4 Inspect all glassware before use and remove from service any glassware that is chipped or broken.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm (TWA)	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness, and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.

(1) Always add acid to water to prevent violent reactions.
(2) Exposure limit refers to the OSHA regulatory exposure limit.

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.1.2 Sample Purger: The recommended purging chamber is designed to accept between 5 mL and 25 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low level soils are purged directly from a VOA vial.

6.1.3 Trap: A variety of traps may be used, depending on the target analytes required. The Vocarb 3000 (K Trap Supelco) is recommended. Other traps such as O.I. #10 (Tenax / Silica gel / Carbon Molecular Sieve), EST Proprietary E10, or Vocarb 4000 may be used if the Quality Control criteria are met.

6.1.4 Desorber: The desorber should be capable of rapidly heating the trap up to 250 °C depending on the trap packing material. Many such devices are commercially available.

6.1.5 Sample Heater: A heater capable of maintaining the purge device at 40 °C is necessary for low level soil analysis.

6.1.6 Purge-and-trap and Autosampler: An autosampler capable of sampling from a sealed vial. An EST Centurion coupled with an EST Evolution purge and Trap or a Varian Archon coupled with an OI purge and trap are most commonly used.

6.1.7 Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.

6.1.8 Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.1.8.1 Column 1: 30 m X 0.25 ID Rxi-624Sil MS with 1.4 um film thickness. This is the most common one used.

6.1.8.2 Column 2: 60 m X 0.25 ID DB-624 with 1.4 µm film thickness.

6.1.8.3 Column 3: 75 m X 0.53 ID DB-624/624.1 wide bore with 3 µm film thickness.

6.1.9 Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 amu every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-bromofluorobenzene (BFB) on column are injected onto the gas chromatograph inlet.

6.1.10 GC/MS interface: Any interface (including direct introduction to the mass spectrometer) that achieves all acceptance criteria may be used.

6.1.11 Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. In addition, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Microsyringes: 10 µL and larger, 0.006-inch ID needle.

6.3.2 Syringe: 5 or 25 mL glass with Luerlok tip, if applicable to the purging device.

6.3.3 Balance: Analytical balance capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.3.4 Vials: 2 mL, 20 mL, and 40 mL with screw caps and Teflon liners

6.3.5 Disposable magnetic stirrers for low-level soil analyses

6.3.6 Volumetric flasks: 10 mL, 50mL and 100 mL, class A with ground-glass stoppers.

6.3.7 Spatula: Disposable wooden tongue depressors.

6.3.8 Disposable pipettes: Pasteur.

6.3.9 pH paper: Wide range.

6.3.10 Gases:

6.3.10.1 Helium: Ultra high purity, grade 5, 99.999% (carrier gas).

6.3.10.2 Compressed nitrogen: Used for instrument pneumatics and sample purge.

7.0 Reagents and Standards

7.1 Methanol: Purge and Trap Grade, High Purity

7.2 Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See Section 9.4.) Reagent water may be purchased as commercial distilled

water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable including boiling reagent water followed by Nitrogen gas purging.

7.3 Sand: Reagent grade Ottawa sand or equivalent.

7.4 Antifoam B, Silicon Emulsion, J. T. Baker, 100% purity.

7.5 Sodium bisulfate ($NaHSO_4$), reagent grade

7.6 If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers' expiration date.

7.7 Calibration Stock Standard Solutions

Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10 to -20 °C. Stock standards for gases must be monitored frequently and replaced within one month after opening or sooner depending on the manufacturer's expiration date. Secondary working standards and aliquots for gases must be replaced within one week after opening. Other stock standards must be replaced at least every 6 months.

7.8 Calibration Working standards

A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20%, then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.9 Aqueous calibration standards are prepared in reagent water using the secondary dilution standards. Standards such as continuing calibration verification standards (CCV) and closing calibration verification standards (CCVC) are to be prepared daily.

7.10 Internal standards (IS) are added to all samples, standards, and blank analyses either by the auto sampler or manually by an analyst. Refer to Tables 8 and 8A for internal standard components.

7.11 Surrogate Standards: Refer to Tables 9 and 9A for surrogate standard components and spiking levels.

7.12 Laboratory Control Sample Spiking Solutions: A second source set of standards is used and contains the Main_B, Gas/Ket_B, and SS-2-Cleve. Compounds not included in these standards can be spiked with lab pre-approval. See TALS Reagent Module for these standards for complete list of target analytes.

7.13 Matrix Spiking Solutions: The matrix spike contains the same components as the LCS.

7.14 Tuning Standard: The IS/SURR standard used by the auto sampler is used and will deliver 5-50 ng of BFB on column upon injection. Introduction of BFB into the column is most commonly performed by the Purge and Trap system but can also be manually introduced into the GC inlet by the analyst as long as the amount injected is 5-50 ng on column.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Water samples

8.1.1 Water samples are collected in triplicate in 40 mL glass VOA vials with PTFE-lined septum caps with minimal headspace. There should be no bubbles present in the container larger than ~6 mm.

8.1.1.1 Analysts will select the VOA vial without a red sticker for analysis if possible. If all vials contain red stickers then the VOA vials with the smallest headspace/bubbles will be chosen for analysis. Any VOA vials analyzed with a red sticker (headspace >6mm) will be documented in an NCM.

8.1.2 Preservation depends upon the target analytes and the sampling location. At a minimum, aqueous samples are stored refrigerated at ≤ 6 °C and not frozen. Specific preservation requirements are given in the following table. If multiple analytes are requested, it may be necessary to provide aliquots with different preservations. For each preservation technique, the samples should be collected in triplicate.

8.1.3 SW-846 states that if carbonaceous materials are present, or if MTBE and other fuel oxygenate ethers are present and a high temperature sample preparative method is to be used, do not acid preserve the samples. The holding time for these unpreserved samples is 7 days. SW-846 does not otherwise provide guidance for processing unpreserved samples. EPA MICE has interpreted the holding time on an unpreserved sample as 7 days.

Preservation and Holding Time for Volatiles in Water

Analyte(s)	Reference	Preservation	Holding time	Dechlorination Required[1]
Routine target analytes[2]	SW-846, Ch. 4	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	SW-846, Ch. 4	Cool, ≤6ºC	7 days	Y
	624/624.1	Cool, ≤6ºC, pH < 2 with 1:1 HCl	14 days	Y
	624/624.1	Cool, ≤6ºC	7 days	Y
Acrolein[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC (no HCl)	3 days	Y
	40 CFR Part 136 (624/624.1)	Cool, ≤6ºC, pH 4-5	14 days	Y
Acrylonitrile[3]	SW-846, Ch. 4	Cool, ≤6ºC, pH 4-5	7 days	N

Analyte(s)	Reference	Preservation	Holding time	Dechlorination Required[1]
	40 CFR Part 136 (624/624.1)	Cool, ≤6°C (no HCl)	14 days	Y
	40 CFR Part 136 (624/624.1)	Cool, ≤6°C, pH 4-5	14 days	Y
2-Chloroethylvinyl ether (2-CLEVE)[4]	SW-846, Ch. 4	Cool, ≤6°C (no HCl)	7 days	Y
	624/624.1	Cool, ≤6°C (no HCl)	14 days	Y

[1] If residual chlorine is present, 2 drops of 10% sodium thiosulfate are added. *Never add acid preservative directly to a dechlorinating agent prior to sample collection.*

[2] Separate aliquots must be collected and preserved as indicated if acrolein, acrylonitrile, 2-CLEVE (by Methods 8260B/C/D or 624/624.1), vinyl chloride (by Method 8260B/C/D) or styrene (by Method 8260B/C/D) are also to be analyzed. If aromatic and biologically active compounds are analytes of interest, acid preservation is necessary.

[3] SW-846 only recommends a 7 day holding time for acrolein and acrylonitrile preserved to pH 4-5. As this preservation is difficult to achieve with HCl, the laboratory recommends unpreserved aliquots with a three day holding time.

According to 40 CFR Part 136, the preferred method for acrolein and acrylonitrile is Method 603. In the Method Update Rule published in the Federal Register on May 18, 2012 (40 CFR Parts 136) EPA approved Method 624/624.1 for the determination of acrolein and acrylonitrile in wastewater. The current sample preservation and holding time requirements for acrolein and acrylonitrile apply to these compounds when analyzed by Method 624/624.1. Implementation of this rule is subject to individual state program decisions and timetables.

[4] SW-846 includes vinyl chloride and styrene in the list of compounds which require unpreserved sample for analysis. Method 624/624.1 does not include these two analytes on the standard analyte list. The laboratory recommends that if these analytes are requested by Method 624/624.1 that unpreserved aliquots be submitted.

8.2 Soil Samples

8.2.1 Sampling Techniques and Containers

8.2.1.1 Soil samples can be taken using the EnCore™ sampler. Typically three Encores are collected per sampling location. At specific client request, unpreserved soil samples may be accepted for preservation at the lab.

8.2.1.2 The more common way to collect soils is with Terra Core kits. Typically three aliquots are collected. Terra Core kits consist of the Terra Core sampling device and three 40 mL tared VOA vials. Samples are extruded into empty tared vials and preserved at the laboratory or are extruded into pre-preserved, tared vials in the field and transported to the laboratory for analysis.

8.2.2 An additional bottle of unpreserved soil for each sampling location must be shipped for percent moisture determination.

8.2.3 A second bottle of unpreserved soil is sent for screening.

8.2.4 Unpreserved Soils

8.2.4.1 At specific client request unpreserved soils packed into glass jars or brass tubes may be accepted and sub sampled in the laboratory. This is the old procedure based on Method 5030A. It is no longer included in

subsequent revisions of Method 5030 and is likely to generate results that are biased low, possibly by more than an order of magnitude.

8.2.4.2 The maximum holding time is 14 days from sampling until the sample is analyzed. Unpreserved samples should be analyzed as soon as possible. The lack of preservation should be addressed in the case narrative.

8.3 Trip blanks, consisting of laboratory prepared water samples with acid preservative, are also provided when bottles are supplied by the laboratory to the field. Trip blanks are used for both water and soil samples to monitor potential contamination from volatile compounds in transit and in the field.

8.4 A holding blank is stored in each refrigerator with the samples. This is analyzed every 14 days (see SOP DV-QA-0013).

8.5 Preservation and holding times for volatiles in soils are summarized in the following table, based on SW-846 Method 5035A.

Preservation and Holding Time for Volatiles in Soil
Method 5035A

Sample Handling in Field	Preservation[1,2,3]	Holding Time
Sample Extruded into Empty Vial in Field, sealed and shipped to Lab	Cool to ≤6°C or freeze on site to -7°C in field (do not freeze below -20°C), maintained cold in transit Upon receipt the laboratory must: a) Maintain the sample frozen or b) Add 5 mL water and freeze (-20°C to -7°C), or c) Add 5 mL water and 1 g $NaHSO_4$, store at ≤6°C or d) Add methanol, store at ≤6°C	48 hours from sampling to other preservation 14 days from sampling
Encore sampler used for transport	Cool to ≤6°C or freeze on site to -7°C in field (do not freeze below -20°C), maintained cold in transit Upon receipt at laboratory sample is extruded to a sealed vial and either frozen to <-7°C or chemically preserved (see above).	48 hours from sampling to other preservation 14 days from sampling
Field Preserved	Sample is extruded into a vial containing reagent water and frozen on –site to <-7°C	14 days
Field Preserved	Sample is extruded into a vial containing reagent water and cooled on –site to ≤6°C for 48 hours or less then frozen to <-7°C upon receipt at laboratory	14 days if samples frozen within 48 hours
Field Preserved	Sample is extruded into a vial containing reagent water and 1 g NaHSO4 and cooled to ≤6°C. Maintained ≤6°C upon receipt at laboratory.	14 days

Field Preserved	Sample is extruded into a vial containing methanol and cooled to ≤6°C. Maintained ≤6°C upon receipt at laboratory.	14 days

[1] If multiple aliquots of sample are provided a combination of preservation techniques may be used

[2] Reactive compounds such as 2-chloroethylvinyl ether readily break down under acidic conditions. If these types of compounds are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.

[3] For biologically active soils, immediate chemical or freezing preservation is necessary due to the rapid loss of BTEX compounds within the first 48 hours of sample collection.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. Use the LIMS Method Comments to determine specific QC requirements that apply when processing samples in the laboratory.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Proper cleaning of laboratory glassware is an essential element of contamination control and therefore an important element of the laboratory's quality control program. Proper cleaning of glassware is reflected in acceptable method blanks results.

9.2.1 Gastight syringes must be triple rinsed with Methanol and air dried.

9.2.2 Class A volumetric flasks must be triples rinsed before and after each use and air dried. **Note:** Do not bake or use wire brushes to clean Class A glassware.

9.3 Batch Definition

Batches are defined at the sample preparation stage. The batch is a set of up to 20 samples of the same matrix, plus required QC samples (method blank, lab control sample, and matrix spike/matrix spike duplicate), processed using the same procedures and reagents within the same time period. Batches should be kept together through the whole analytical process as far as possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. A method blank must be run on each instrument. See Policy DV-QA-003P for further details.

9.4 Method Blanks

For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles in water, the method blank consists of reagent water. For low-level volatiles in soil, the blank medium is Ottawa sand preserved with DI water or Sodium Bisulfate depending on how the samples are preserved. For medium-level volatiles, the method blank consists of Ottawa sand to which surrogates are added and extracted with methanol. The method blank is carried through the entire preparation and analytical procedure.

NOTE: If both DI and sodium bisulfate preserved samples are analyzed the QC samples should be prepared with sodium bisulfate.

Acceptance Criteria: The method blank must not contain any analyte of interest at or above one-half the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

For DOD QSM 4.2 or 5.0, common lab contaminants must not exceed the reporting limit.

The method blank must have acceptable surrogate recoveries. (See Section 9.5)

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Actions: If the analyte is a common laboratory contaminant (i.e., methylene chloride, acetone, 2-butanone, and carbon disulfide), the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.

Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the associated samples.

If there is no target analyte greater than one-half the RL in the samples associated with an unacceptable method blank, the data

may be reported with qualifiers. Such action should be taken in consultation with the client.

If surrogate recoveries in the blank are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B", and appropriate comments may be made in the narrative to provide further documentation.

9.5 Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 8A.

For DoD QSM 4.2 or 5.0, special method codes in TALS include program specific QC limits for surrogates. If limits are not available in the appropriate QSM, in-house historical limits are to be used.

Acceptance Criteria: Acceptance limits for surrogate recoveries are set at ± 3 standard deviations around the historical mean. Surrogate recovery limits are updated annually and stored in the LIMS.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Actions: If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Re-prepare and reanalyze the sample or flag the data as “Estimated Concentration” if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is necessary to re-prepare/reanalyze a sample only once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and re-preparation/reanalysis is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.6 Laboratory Control Samples (LCS)

An LCS is analyzed for each batch. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS spiking solution is prepared from a different source than are the calibration standards. See Section 7.12 for description of spiking solution. For low-level volatiles in soil, the LCS medium is Ottawa sand preserved with DI water or sodium bisulfate depending on how the samples are preserved. For medium-level volatiles, the LCS consists of Ottawa sand to which surrogate and LCS spiking solutions are added and extracted with methanol. The LCS is carried through the entire preparation and analytical procedure. If antifoam agent is added to samples it must also be added to the LCS.

NOTE: If both DI and sodium bisulfate preserved samples are analyzed the QC samples should be prepared with sodium bisulfate.

NOTE: DoD and South Carolina require that all target compounds are spiked and reported. Specific reporting requirements for spiked compounds must be in the method comments for the job. Each project must be set up to include all spiked compounds to be reported. (NELAP requires that all targeted compounds be spiked across a two year period in the MS/MSD.)

Acceptance Criteria: The LCS recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. An LCS that is determined to be within acceptance criteria effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch. Recovery limits are updated annually and stored in the LIMS. For DoD QSM 4.2 or 5.0, special method codes in TALS include program specific QC limits. If limits are not available in the appropriate QSM, in-house historical limits are to be used.

If there are a large number of analytes in the LCS, then a specified number of results may fall beyond the LCS control limit (3 standard deviations), but within the marginal exceedance (ME) limits, which are set at ± 4 standard deviations around the mean of historical data. Marginal exceedances are recognized and allowed by NELAP. DoD QSM 5.0 allows marginal exceedances but excludes them from being applied to specified critical compounds of concern. Marginal exceedances must not be applied to an analyte specified by the project team as a compound of concern.

The number of marginal exceedances is based on the number of analytes in the LCS, as shown in the following table:

# of Analytes in LCS	# of Allowed Marginal Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

If more analytes exceed the LCS control limits than is allowed, or if any analyte exceeds the ME limits, the LCS fails and corrective action is necessary. Marginal exceedances must be random. If the same analyte repeatedly fails the LCS control limits, it is an indication of a systematic problem. The source of the error must be identified and corrective action taken.

Note: Additional criteria are stated in the North Carolina QAS.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Note: Some programs (e.g., South Carolina) do not allow marginal exceedances. Please see the QAS's in the public folders for the current requirements.

Note: DoD allows marginal exceedances except for critical compounds of concern. These must be specified in the communication of project requirements to the lab.

Corrective Actions: If any analyte or surrogate is outside established control limits as described above, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and reanalysis of the batch.

If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. Examples of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be reviewed and documented with the client before reporting.

If samples are ND and LCS recovery for an analyte is out high, the sample may be reported with a qualifier, if acceptable to the client.

If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all

associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.7 Matrix Spike and Matrix Spike Duplicate (MS/MSD)

For each QC batch, analyze a matrix spike and matrix spike duplicate. See Section 7.13 for description of spiking solution. The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

NOTE: DoD and South Carolina require that all target compounds are spiked and reported. Specific reporting requirements for spiked compounds must be in the method comments for the job. Each project must be set up to include all spiked compounds to be reported. (NELAP requires that all targeted compounds be spiked across a two year period in the MS/MSD.)

Acceptance Criteria: The MS/MSD recovery for the control analytes must be within established control limits. Unless otherwise specified in a reference method or project requirements, the control limits are set at ± 3 standard deviations around the mean of the historical data. The relative percent difference (RPD) between the MS and the MSD must be less than the established RPD limit, which is based on statistical analysis of historical data. MS/MSD recovery and RPD limits are updated annually and stored in the LIMS.

DoD QSM 5.0 requires the RPD limit to be ≤ 20%. DoD MS/MSD recovery limits are the same as LCS limits. See Section 9.6.

Note: Only use data associated with acceptable Calibration and QC data for 624/624.1 compliance samples.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- When the parent sample and MS/MSD concentration are above the linear range; the MS/MSD and parent sample **MUST** be re-analyzed at a dilution.
- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);

- Flag the data for any results outside of acceptance limits and note it on the final report.

- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this request.

NOTE: A sample duplicate is not performed as precision is obtained from either the MS/MSD or LCS/LCSD pair. Use of the MS/MSD precision is preferred as not all samples will contain measurable concentrations of target analytes. Any samples that have target analytes at such low levels do not provide useful precision data.

9.8 Acid Preservation or pH adjustment

According to the source methods, the stability of 2-chloroethylvinyl ether, acrolein, and acrylonitrile is reduced when subjected to low pH. It is therefore not recommended that these compounds be analyzed routinely from preserved VOA vials and since there is no reasonable way to achieve pH between 4 and 5, it is recommended that unpreserved vials be used for the analysis of these compounds.

Acceptance Criteria: To ensure detection of these compounds, samples must be processed correctly. Where Method 624/624.1 is being used for compliance purposes, the regulatory hold times take precedence.

Corrective Actions: If 624/624.1 data are not being generated for compliance purposes, the technical stability of the compounds may be considered. Where method 8260 is the base method, it is allowable to qualify the results as estimated. To deviate from the regulatory hold times, the following documentation must be maintained:

- A NCM must be generated by the lab that the samples are for non-compliance.
- A NCM must be generated that results are not method compliant.
- A NCM must be generated if the samples are not analyzed from an upreserved container.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more

detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preservation using EnCore™ Samplers.

10.3.1 Preservation in Methanol (Medium-Level Analysis)

10.3.1.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it in the prep batch. Select the correct cell in prep batch worksheet and once the balance is stable hit the print key. The weight will automatically be recorded in the batch record. Quickly add 5 mL of methanol and cap the vial.

10.3.1.2 If sufficient samplers are provided (or for the sample(s) designated by the client), prepare MS and MSD samples as above.

10.3.1.3 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials. Add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.3.1.4 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.3.2 Preservation in Water (Low-Level Analysis)

10.3.2.1 Extrude the (nominal) 5 g sample from one of the Encore™ samplers into a tared 20 mL VOA vial. Obtain the weight of the soil added to the vial and record it in the prep batch. Select the correct cell in prep batch worksheet and once the balance is stable hit the print key. The weight will automatically be recorded in the batch record. Quickly add 5 mL of water and a magnetic stirrer. Cap the vial. Repeat for the remaining aliquot.

10.3.2.2 If requested by the client, 1 g of sodium bisulfate is added to the second sample preserved with water.

10.3.2.3 If sufficient samplers are provided for a sample in the batch, or for any samples identified by the client, prepare MS and MSD samples as above.

10.3.2.4 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into separate, tared 20 mL VOA vials.

Add 5 mL of water to the MB and LCS. Add a magnetic stirrer. Cap tightly. Store with the samples.

10.3.2.5 After all of the samples in the prep batch have been weighed and preserved in water the status of the samples and QC in the prep batch will remain at "batched" status for backlog tracking purposes.

10.3.2.6 When the samples are loaded on the instrument for analyses then the analyst can change the status of the samples and QC to "2nd level" so that the prep method's status will change from "Ready" to "2nd level" therefore changing the status of the analytical method from "wait" to "Ready"

10.3.2.7 Store the samples and QC samples in the freezer until screening is performed.

10.3.3 Screen the samples. (See Section 10.5.) If the screen indicates any samples will be analyzed as medium level, go to Section 10.5.1. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.8.

10.4 Sample Storage for Field Preserved Samples

10.4.1 Obtain the weight of the soil added to each vial and record it in TALS. The samples will arrive with a tare weight (weight of vial, preservative and stir bar) and no other labels attached to it. The unlabeled vial is weighed and the difference between this weight and the tare weight is the weight of the sample used for analyses.

NOTE: Do not affix label to unprepared/unweighed sample vial.

10.4.1.1 Prepare a method blank and LCS sample by weighing approximately 5 g of baked Ottawa sand for each into tared VOA vials for each analysis method. The low level preparation is done in a 40mL VOA vial and the medium level preparation is done in a 20mL VOA vial.

10.4.1.1.1 For the medium level method, in a 20mL VOA vial add 5 mL of methanol to the blank. For the LCS, the volume of methanol added is dependent upon the spike list. Add 4.95 mL methanol if the Short List is to be spiked and 4.85 mL methanol if the full list is to be spiked. Cap tightly. Store with the samples.

10.4.1.1.2 For the low level method in a 40ml VOA vial add water instead of methanol using the same volumes as in Section 10.4.1.1.1.

10.4.1.2 Store the samples and QC samples in the freezer until screening is performed. Surrogates and LCS/MS/MSD spikes are only added if it is determined the samples will be analyzed at the medium level.

10.4.2 Screen the samples. (See Section 10.5) If the screen indicates any samples will be analyzed as medium level, go to Section 10.7.5. If the screen indicates any samples will be analyzed as low level, go to Section 10.7.8.

10.5 Sample Screening

10.5.1 Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. See SOP DV-MS-0009. Alternatively, an appropriate aliquot can be determined from sample histories.

10.6 Sample Preparation for Medium-Level Analysis – Field or Lab Preserved

10.6.1 For each of the samples that are determined to be Medium-Level samples by the screening procedure, add the correct amount of surrogate spiking mixture for a final concentration of 2 µg/mL. Example: 4 µL of 2500 µg/mL for a nominal 5 g sample or 20 µg/mL for a nominal 25 g sample. Cap the sample vial. Surrogates are added to all QC samples as well as field samples.

10.6.2 Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples for a final concentration of 2 µg/mL.

10.6.3 Add the correct amount of matrix spiking solution to the LCS sample for a final concentration of 2 µg/mL. If 25 g samples are being used, adjust the proportions for the LCS accordingly.

10.6.4 Shake the samples for two minutes to distribute the methanol throughout the soil.

10.6.5 Centrifuge the samples to clarify the extract.

10.6.6 Remove a portion of methanol and store in a clean Teflon-capped vial with no headspace refrigerated at $\leq$ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

10.7 Sample Analysis Procedure

10.7.1 All analysis conditions for samples must be the same as for the initial and continuing calibration standards (including purge volume, time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

10.7.2 All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a method blank, an LCS, and a MS/MSD.

10.7.2.1 If there is insufficient time in the 12-hour 624/624.1 period to analyze 20 samples, the batch may be continued into the next tune period. The 12-hour tuning requirements in Section 10.7.12.3 and 12-hour continuing calibration requirements in 10.7.14 must still be met. However, if any re-tuning or recalibration of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new QC batch must be started. For high-level soils the batch is defined at

the sample preparation stage. Laboratory generated QC samples (Blank, LCS, MS/MSD) do not count towards the maximum 20 samples in a batch. Field QC samples are included in the batch count.

10.7.2.2 Any reruns must be part of a valid analytical batch. If dilutions of sample are analyzed in the same 12-hour tune they do not count towards the maximum batch count. (See DV-QA-003P.)

10.7.3 Water Samples

10.7.3.1 Purge-and-trap units that sample from a VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.3.2 All samples and standard solutions must be at ambient temperature before analysis.

NOTE: Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

10.7.4 Preparation of water samples for analyses on EST Centurion/Evolution setup

10.7.4.1 Calibration standards are prepared in 50ml Class A volumetric flasks and quickly transferred to clean 40-mL VOA vials.

10.7.4.2 For Method 8260B, EPA624.1, and EPA 8260C, 5mL sample aliquots are sampled by the auto sampler unless dilutions are performed. Unless a sample requires an external dilution, then sample containers will not be opened and they will be placed on the Centurion auto sampler for analysis. Dilutions can be performed by the auto sampler in the following increments (2,4,5,10,20,40,50,100,200 and 400:1). Dilutions higher than the ones mentioned may require a sample vial to be opened. Preparing an MS/MSD may require a sample vial to be opened.

10.7.4.3 If the dilution required would use less than 5 µL of sample, then serial dilutions must be made in volumetric flasks.

10.7.4.4 Check the pH of the sample by dipping the pH paper into the sample that is remaining in the VOA vial after the auto sampler has finished taking the aliquot for analysis (the remaining sample is not used in analysis.) Document the pH value on the run log. If the pH is not as expected, based on the sample type and preservation, document in an NCM in the LIMS.

10.7.4.5 Sample remaining in the vial after sampling is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

10.7.4.6 For TCLP samples, use 5.0mL of TCLP leachate. Bring to a volume of 50mL with reagent water in a Class A volumetric flask. Pour sample into a 40mL vial and analyze.

10.7.4.7 Surrogates and internal standards are added to each sample at the instrument at the time of purging.

10.7.4.8 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe needle through the septum into the water. Surrogates and internal standards are added to these samples by the instrument.

10.7.4.9 Purge the sample for eleven minutes (the trap should be below 50 °C).

10.7.4.10 After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 2-5 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

10.7.4.11 Desorb time, bake time, and temperature are optimized for the type of trap in use.

NOTE: The same conditions must be used for samples and standards.

10.7.4.12 If foaming of the sample occurs, reanalyze the sample with the addition of 1 µL of an antifoaming agent such as Antifoam B (J. T. Baker). A method blank spiked with 1 µL of the Antifoam B must also be analyzed with the sample. Make notation of "AF" on runlog for each sample to which antifoam is added.

10.7.5 Methanol Extracts of Soils

10.7.5.1 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations (typically 20 mL).

10.7.5.2 Add no more than 200 µL of methanolic extract (from Section 10.3.1 or 10.5.1) to the syringe for each sample and QC sample.

10.7.5.3 Calibration standards and spiking solutions are added to the CCVs, LCS and MS/MSD samples by the analyst prior to purging by inserting the syringe through the septum of the vial.

10.7.5.4 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe.

10.7.5.5 Only internal standards are added at the instrument for methanol extracts.

10.7.5.6 Load the sample onto the purge and trap device and analyze as for aqueous samples. (See Section 10.7.3.)

10.7.6 Preparation of water samples for Centurion/Evolution setup

10.7.6.1 Standards

10.7.6.1.1 All standards are prepared in 50mL volumetric flasks

10.7.6.1.1.1 Fill volumetric 90% full with DI water, spike with standard(s), bring to volume with DI water squirt bottle and invert 3 times to mix. Pour into a labeled 40mL VOA vial without headspace. Place full VOA vial in auto sampler tray.

10.7.6.1.1.2 Volumetric glassware must be cleaned with DI water after use and inverted in rack to dry.

10.7.6.2 Samples

10.7.6.2.1.1 Scan the bar code from the sample vial into the instrument work list and the sequence. Place the vial in the correct position in the auto sampler try. If the sample requires dilution see the auto sampler manual for instructions on setting up the sequence.

10.7.6.2.1.2 After the analysis is complete the pH can be taken from the left over sample in the vial and noted on the sequence print out. The left over sample in the vial is not to be used for re-analyses.

10.7.7 Liquid Wastes that are Soluble in Methanol and Insoluble in Water

10.7.7.1 Pipette 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

10.7.7.2 Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly.

10.7.7.3 For an MS/MSD pair, add 6 mL of methanol to 2 mL of the sample in a tared vial. Add 1 mL of surrogate solution and 1 mL of matrix spike solution.

10.7.7.4 Prepare an LCS by adding 1 mL of surrogate solution and 1 mL of matrix spike solution to 8 mL of methanol.

10.7.7.5 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic-free water as used in the calibrations.

10.7.7.6 Add no more than 200 µL of methanolic extract (Section 10.7.7.2) to the syringe. Add internal standard (if used).

10.7.7.7 Load the sample onto the purge and trap device and analyze as for

aqueous samples using 5 mL reagent water.

10.7.7.8 If less than 5 µL of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 5 µL will be added to the water in the syringe. (See Section **Error! Reference source not found.**.)

10.7.8 Low-Level Soil Sample Analysis following SW846 Method 5035A

10.7.8.1 This technique is to be used when samples are collected utilizing SW-846 Method 5035A. Pre-weighed vials are used to collect approximately a 5 gram aliquot of soil (see section 8.2).

10.7.8.2 The sample vial should not have any labels applied to it until after the sample weight has been taken. Any labels applied prior to weighing will affect the initial weight calculation of the sample, because it was not accounted for in the tare weight.

10.7.8.3 Purge-and-trap units that sample from the VOA vial should be equipped with a module that automatically adds surrogate and internal standard solution to the sample prior to purging the sample.

10.7.8.4 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial.

10.7.8.5 The autosampler will heat and stir each sample as it is purged.

10.7.8.6 If any target analytes exceed the calibration range, analysis of the methanol preserved sample must be performed.

10.7.9 Low-Level Solids Analysis When Field Samples are Provided in a Jar

NOTE: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.

10.7.8.1 This method is based on purging a heated sediment/soil sample mixed with water and, if applicable, internal and matrix spiking standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40 °C.

10.7.8.2 Do not discard any supernatant liquids. Mix the contents of the container with a disposable tongue depressor as spatula.

10.7.8.3 Weigh out 5 g (or other appropriate aliquot) of sample into a clean VOA vial. Record the weight to the nearest 0.1 g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 1.0 g. If the sample is contaminated with analytes such that a purge amount less than 1.0 g is appropriate, use the medium-level

method described in Section 10.7.5 with preparation described in Section 10.5.1.

10.7.8.4 Rinse a 5 mL gas-tight syringe with organic-free water, and fill. Compress to 5 mL. Inject the spiked water into the VOA vial that contains the soil sample and add a stirring bar.

10.7.8.5 The above steps should be performed rapidly and without interruption to avoid loss of volatile organics.

10.7.8.6 Prepare a Method Blank and LCS using 5 g of Ottawa sand and 5 mL of water. Add a stirring bar to each. Prepare the MS/MSD (based on the sample requested by the client. The LCS spiking solution is added via a syringe inserted through the septum of the vial to the LCS and MS/MSD samples.

10.7.8.7 Low level soil samples may be analyzed with a 1 g aliquot in place of the 5 g aliquot, mixed with water. If higher dilutions are required, the methanol extract (medium level) will be analyzed.

10.7.8.8 Surrogate and internal standards are added automatically to all samples at the instrument.

10.7.8.9 The autosampler will heat and stir each sample as it is purged.

10.7.8.10 Soil samples that have low internal standard recovery when analyzed (< 50%) should be reanalyzed once to confirm matrix effect.

10.7.9 Initial Review and Corrective Actions

10.7.9.1 If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minute from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required. DoD QSM 5.0 requires that the retention time for any internal standard be within 10 sec of the midpoint of the ICAL. If accepted by the client, this window can be expanded to 30 sec of the midpoint of the ICAL.

10.7.9.2 If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

Sample internal standard areas are compared to the mid-point of the supplemental initial calibration internal standard areas. Responses from 50% to 200% are acceptable. If a sample fails to meet these internal standard criteria, further investigation is necessary. If the change in sensitivity is a matrix effect confined to an individual sample, reanalysis is not necessary. If the change in sensitivity is due to

instrumental problems, all affected samples must be reanalyzed after the problem is corrected.

10.7.9.3 The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

10.7.10 Dilutions

10.7.10.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the sample or extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.7.10.2 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment.

10.7.10.3 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.7.11 Instrument Set-up

Prior to the analysis of samples and blanks, the GC/MS system must be tuned and calibrated. Tuning is accomplished by analyzing 4-bromofluorobenzene (BFB) to establish that the GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations to determine the linearity of the response utilizing target calibration standards. The calibration must be verified each twelve-hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.7.12 Recommended Instrument Conditions

10.7.12.1 General

Parameter	Setting
Electron Energy:	70 volts (nominal)
Mass Range:	35–300 amu
Scan Time:	to give at least 5 scans/peak, ≤ 2 second/scan
Injector Temperature:	200 – 250 ˚C
Source Temperature:	According to manufacturer's specifications
Transfer Line:	Temperature: 250 – 300 ˚C
Purge Flow:	40 mL/minute
Carrier Gas Flow:	1-15 mL/minute, dependent upon column specifications

10.7.12.2 Gas Chromatograph Suggested Temperature Program

The following temperature programs vary with the column type used.

BFB Analysis

Initial Temperature:	150 ˚C
Initial Hold Time:	0.00 minutes
1st Temperature Program:	50.00 ˚C/minute
Final Temperature:	220 ˚C
Final Time:	4.00 minutes
2nd Temperature Program:	OFF
Post Temperature:	0 ˚C
Post Time:	0.00 minutes
Run Time:	5.40 minutes

Sample Analysis

Initial Temperature:	40 ˚C
Initial Hold Time:	1 minute
1st Temperature Program:	15 ˚C/minute
Final Temperature:	220 ˚C
Final Hold Time:	0.5 minutes
GC inlet split ratio	30:1 or higher

10.7.12.3 Instrument Tuning

Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng on column injection of BFB. Analysis must not begin until these criteria are met. These

criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.7.13 Initial Calibration

10.7.13.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-S-005, *Calibration Curves (General)* and in the public folder *Arizona Calibration Training.*

10.7.13.2 A series of five or more initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Certain analytes are prepared at higher concentrations due to poor purge performance. The following calibration curves are maintained. Calibration levels for each analyte are given in the stated tables. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument or program requirements.

Initial Calibration by Matrix and Method

Method	Matrix	Purge Volume	Calibration Levels
624/624.1/8260C/D	Water	5 mL	Table 5A
8260B	Water	5 or 20 mL	Tables 5 and 5A
8260B/C/D	Soil (low level)	5 mL	Tables 4 and 4A
8260B/C/D	Soil (Methanol Extract)	20 mL reagent water + 100 µL Methanol	Tables 6 and 6A
Alaska	Soil	See Appendix B	See Appendix B

10.7.13.3 Calibration levels below the reporting limit may be removed provided that there is a minimum of five calibration points for linear regression and six calibration points for second order calibration. The lowest standard used in the calibration must be at or below the TestAmerica reporting limit.

10.7.13.4 The same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.7.13.5 It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for some tests.

10.7.13.6 Internal standard calibration is used. The internal standards are listed in Tables 8 and 8A. Target compounds should reference the nearest internal standard. Each calibration standard is analyzed and the

response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See Equation 1, Section 11.5.1.1, for calculation of response factor.

10.7.13.7 When setting up initial calibration, surrogate and ICIS calibrations will be performed only on an ICAL containing gases and 2-CLEVE or an ICAL containing the Main compounds. Supplemental list calibrations will be performed separately.

10.7.13.8 Evaluation of retention times

The retention time of each target analyte in each calibration standard should agree within 0.5 min.

10.7.13.9 The % RSD of each of the calibration check compounds (CCC) must be less than or equal to 30%. Refer to Table 12. See Table A-2 for Method 624/624.1 criteria.

10.7.13.10 The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.7.13.11 If the software in use is capable of routinely reporting curve coefficients for data validation purposes and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If it appears that substantially better accuracy would be obtained using quantitation from a curve then the appropriate curve should be used for quantitation. The correlation coefficient (coefficient of determination for non-linear curves) must be ≥ 0.990.

NOTE: Additional criteria are stated in the North Carolina QAS. South Carolina does not allow the use of second order curves.

10.7.13.12 If the software in use is capable of routinely reporting curve coefficients for data, and if the average of all the %RSDs in the calibration is > 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response. Otherwise, the correlation coefficient, r (coefficient of determination, r^2 for non-linear curves) must be ≥ 0.995 (i.e., $r^2 \geq 0.99$). DoD QSM 5.0 requires that the correlation coefficient for linear fits be ≥ 0.995 (i.e., $r^2 \geq 0.99$).

NOTE: Some states (like Arizona) and federal programs do not allow the use of grand mean. Refer to the Arizona QAS and SOP DV-QA-024P.

10.7.13.13 Once the initial calibration has been evaluated and determined to be

valid, the calibration must be verified with an Initial Calibration Verification (ICV) using a standard prepared from an alternate source. All compounds in the ICV must be <35% drift when compared to the initial calibration, except poor performers (see Table 15) which must be <55% drift. The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against spike recovery data and method performance data, where available, to validate each compound as a "poor performer." The ICV is generally run at the same concentration as the level 5 standard. See Table A-2 for method 624/624.1 criteria.

DoD QSM 5.0 requires the ICV analytes must be within ± 20% of the true value. Poor performers listed in the TestAmerica Technical Specifications may be ± 30% if approved by the client.

South Carolina requires that all analytes of interest in the ICV must recover at ≤ 30% (≤ 40%) for poor purgers prior to sample analysis.

10.7.13.14 If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration, Section 10.7.14.

10.7.13.15 A separate five point calibration must be prepared for analysis of low-level soils. Low-level soils analysis requires the use of a closed vial autosampler. Each standard is prepared by spiking the methanol standard solution through the septum of a VOA vial containing 5 mL of water. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Methanol soil extracts should be analyzed using the methanol calibration curve.

10.7.13.16 Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five point initial calibration. The primary ion for the single standard must generate a peak clearly visible over background noise (greater than three standard deviations at a minimum) and be free of spectral interferences. If the analyte is detected in any of the samples, a five point initial calibration must be generated and the sample(s) reanalyzed for quantitation. However, if the analyte is not detected, the non-detect may be reported and no further action is necessary. A footnote or narrative comment should describe the basis of the reported result.

10.7.14 Continuing Calibration

10.7.14.1 The initial calibration must be verified every twelve hours.

10.7.14.2 Continuing calibration begins with analysis of BFB as described in Section 10.7.12.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 5 calibration standard is used as the continuing calibration standard. See Table A-2 for method

624/624.1 criteria.

10.7.14.3 The RF data from the standards are compared with the initial five-point calibration to determine the percent drift of the CCC compounds.

10.7.14.4 The % drift of the CCCs must be ≤ 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift for most non-CCC analytes must be ≤ 35 %, and for poor performers ≤ 50 % (See Table 15), with allowance for up to six target analytes to have a % drift greater than the applicable limit. For agencies that require specific control limits for non-CCC compounds (i.e., State of Arizona) see Table 14. See Table A-2 for method 624/624.1 criteria.

For South Carolina, the percent drift for non-CCC analytes must be ≤ 30% (≤ 40%) for poor purgers.,

Note: Additional criteria are stated in the North Carolina QAS.

10.7.14.4.1 If none of the CCCs are required analytes, project specific calibration specifications (which may include the use of the CCCs listed in Table 12) must be agreed to with the client.

10.7.14.4.2 Cyclohexanone is unstable in the calibration solution forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists.

10.7.14.5 The retention time of the internal standards in the continuing calibration standard cannot change by more than 30 seconds when compared to the most recent five-point calibration. The internal standard areas must not change by more than a factor of 2 (50 - 200 %) from the mid point standard of the most recent five-point calibration.

10.7.14.6 If the CCCs and/or the SPCCs do not meet the criteria in Sections 10.7.14.3 and 10.7.14.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action, such as a different type of column, will require a new initial calibration.

10.7.14.7 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample desorbed less than or equal to 12 hours after the BFB is acceptable.)

10.7.14.8 Sodium Bisulfate must be added to the CCV when analyzing samples preserved with it.

10.7.14.9 Example analytical sequence for GC/MS volatile analysis:

1. BFB
2. CCV Main
3. CCV Supp
4. LCS
5. LCSD (if required)
6. MB

20 client samples

The above is an example sequence. If any part of lines 1 through 3 does not meet method criteria for running client samples it must be re-analyzed before continuing on to the next step. For example, the CCV(s) can not be run without first passing the BFB tune and LCS/LCSD/MB must follow passing CCV(s).

DoD QSM 5.0 requires a closing CCV be run. The injection time must be within the 12-hour tune time. Acceptance criteria are ± 50%. See Section 10.10 for alternate acceptance criteria if approved for the project.

10.8 Maintenance

See section 20 of the Denver QAM

10.9 Troubleshooting

10.9.8 For RT shifts check purge flow for possible leaks or blockage depending on which way the RT is shifted.

10.9.9 If no peaks appear in chromatogram

10.9.9.1 Check water and standard reservoirs and helium switch is to on position.

10.9.9.2 Check that purge needle is not broken/leaking.

10.9.9.3 Check tune to make sure filaments are still functional.

10.9.9.4 Check GC column for breaks

10.9.9.5 If maintenance was recently performed, re-check that area to make sure all connections are tight.

10.9.10 Samples with high concentrations of hydrocarbons may cause internal standards to fail high in samples analyzed afterwards. Depending on severity either running Methanol blanks or replacing contaminated parts may be needed.

10.9.11 Samples with a basic pH (>10) will cause the surrogate Dibromofluoromethane to fail low.

10.9.12 Samples that contain organic matter may cause internal standard failures.

10.9.13 Temperature variations in the laboratory will cause shifts in the recoveries of water soluble compounds (i.e., Ketones and Alcohols).

10.9.14 If instrument has difficulty passing BFB tune, front end maintenance can solve the problem.

10.9.15 Samples with extreme foaminess can cause issues ranging from failing QC to contamination of instrumentation which may require replacement of contaminated parts.

10.10 Method specific requirements for QSM 5.0

Method Comment Codes and definitions for possible method variances. Method Comment indicates that client has accepted the variance. Requirement if variance NOT accepted is provided in the table below. DoD QSM 5.0 requirements are listed throughout this SOP in the appropriate sections.

Code	If Variance accepted	If Variance not accepted
1SME	Laboratory must be informed of critical compounds of concern to ensure ME not applied to these compounds.	SME is allowed (see table in section 9.5 for number of analytes) except for critical compounds of concern.
2CLC	Common lab contaminants allowed up to RL – Variance identifies those compounds identified as common lab contaminants	Common lab contaminants allowed up to RL. See section 9.4
3HR	Allowed to report biased high failures in CCV/LCS/surrogate when samples ND. Report with NCM and flags.	All QC failures not matrix related must be re-analyzed
4PP	30%D CCV/ICV criteria allowed for poor performers as long as not listed as constituent of concern (COC) by client	Does not recognize poor performers. All targets must pass 20% criteria.
6CCVOrg	If closing CCV fails due to the matrix of samples run prior to it and failure is confirmed once, consult client before reporting	All samples must be bracketed by passing CCV
7MS	Poor performers excluded from 50%D in closing CCV. Re-analyze closing CCV for non-COC's and report others with NCM and flags.	Closing CCV must pass 50%D criteria for all target compounds.
8ISRT	Internal standard window +/- 30 seconds from ICIS	Internal standard window +/- 10 seconds from ICIS

11.0 <u>Calculations / Data Reduction</u>

11.1 Detailed calibration equations can be found in the corporate SOP CA-Q-P-003 "Calibration

Curves and Selection of Calibration Points" and in the public folder, *Arizona Calibration Training*.

11.2 Qualitative Identification

11.2.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library (same library as used for routine sample analysis). Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

11.2.1.1 The sample component retention time must compare to within ± 0.5 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.

11.2.1.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

11.2.1.3 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 %.)

11.2.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

11.2.3 All data are subject to two levels of technical review, as described in SOP DV-QA-0020.

11.3 Tentatively Identified Compounds (TICs)

11.3.1 If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. The following guidelines apply:

11.3.1.1 Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

11.3.1.2 The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

11.3.1.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.3.1.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.3.1.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or co-eluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

11.3.1.6 Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst *assign a tentative identification.*

11.3.2 Chrom setup

Set up for identification of TICs in Chrom is done by using the following settings in the Chrom Method Editor.

Item	Setting
Enable TIC quantitation	Checkmark on
Quant from	Nearest ISTD
Min % for ID	10%
Start and end time	Set both to 0.000
Match Threshold	85%
Disable calibrated TICs	Checkmark on
Enable TIC search	Checkmark on
Path to Library	Must be set to correct path
Subtraction method	Generally best set to no subtraction
Chrom match RT window	Set to 0.1 min

11.4 Data Review

Common Oversights and Possible Fixes

Finding	Fix
Level 4 QC summary report error	8260B/C/D: Set 1,4-Dioxane-d_8 set to “not needed” status in samples and ICAL 624/624.1: Set 1,4-Dioxane-d_8 and TBA-d_9 to “not needed” status in samples and ICAL

Finding	Fix
Missing MS/MSD % recoveries and/or RPD	MS/MSD linked to incorrect parent (all three must be run at same concentration); relink. Missing reagent value – verify and correct on reagent tab. Parent and MS/MSD not processed with "all compounds" list; verify worklist is correct; re-upload if error found in worklist.
Dilution factor/initial volume incorrect	Double check that what is being reported reflects what was performed; correct as needed
Proper sample preservative	8260B/C/D: Create NCM when 2–CLEVE is a target and sample has pH<2. 624/624.1: Create NCM when 2-CLEVE and/or Acrolein is a target and sample pH<2. All: Create NCM when pH does not match preservative type.
QC not in deliverables	All associated QC must be at 2nd level review status to appear in deliverables. This includes any ICIS, ICV, CCVC, LCS/LCSD, MB, LB, and MS/MSD. Use Job Data Review or QC checker to verify and correct as needed.
Incorrect flagging	Re-calculate sample, do not manually remove flag without permission.
False hit for 1,2,3-Trichloropropane	When anti-foam is used take extra care in evaluating any hit for 1,2,3-Trichloropropane. Ensure correct identification.
TIC with large ion 44 early in run	This is usually Carbon Dioxide labeled as an unknown. Verify identification. If reporting TICs it should be marked as undetected in order to not report.

11.4.4 Data Review Procedure

After completion of all analytical data, a first level review is performed. The following items must be scanned in for a 2nd level reviewer to verify: 1) a run sequence log generated from the instrument used for analysis. A stamp must be located in the upper right corner of the run log that has analytical information such as instrument ID, SOP used (including revision number), volume of sample purged, the tune time, and a LIMS batch ID. Relevant information such as data not reported, QC re-analysis, dilutions analyzed, comments on vial headspace and pH of all samples analyzed will be noted on this page. When scanning, it will be the first page to be scanned. 2) A Chrome work list with information about the standards used for all QC and dilution factors used. This will be the second item scanned 3) the SRF sheet which will contain information on dilutions and any analyst bench notes regarding samples including color, viscosity, matrix, and any other pertinent information that the analyst thinks should be included. This will be the 3rd item scanned and 4) a prep batch log for all TCLP, MLE, and low level soil samples. In addition, any prep paperwork for PT samples will be scanned.

11.4.5 Linear Fit

Calibration using least-squares linear regression produces a straight line that does not pass through the origin. The calibration relationship is constructed by performing a linear regression of the instrument response (peak area or peak height) versus the concentration of the standards. The instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The weighting used is the reciprocal of the concentration or the reciprocal of the square of the concentration. The regression produces the slope and intercept terms for a linear equation in the following:

$$R_x = m_1(C_s) + b \qquad \textit{Equation 6}$$

Where:

C_s	=	Analyte concentration in calibration standard, µg/L
R_x	=	Response for analyte
b	=	y - Intercept
m_1	=	Slope

To calculate the concentration in an unknown sample extract, the regression equation is solved for concentration, resulting in the following equations, where C_{ex} is the concentration of the target analyte in the unknown sample:

$$C_{ex} = \frac{[R_x - b]}{m_1} \qquad \textit{Equation 7}$$

Where:

C_{ex}	=	Extract analyte concentration, µg/L
R_x	=	Response for analyte

b = y - Intercept
m_1 = Slope

11.4.6 Evaluation of the Linear Least-Squares Regression Calibration Function:

11.4.6.1 With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations.

11.4.6.2 Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this for weighted regression over the use of an unweighted regression."

Acceptance Criteria:

11.4.6.3 To avoid bias in low level results, the absolute value of the y-intercept must be significantly less than the reporting limit (RL), and preferably less than the MDL.

11.4.6.4 Also examine the residuals, but with particular attention to the residuals at the bottom of the curve. If the intercept or the residuals are large, a second-order regression should be considered.

11.4.6.5 The linear regression must have a correlation coefficient (r) ≥ 0.995 (r^2 ≥ 0.99).

11.4.7 Quadratic Fit

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. The second-order calibration uses the following equation:

$$R_x = m_2(C_s)^2 + m_1(C_s) + b$$ *Equation 8*

Where:

C_s = Analyte concentration in calibration standard, µg/L
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y - Intercept

To calculate the concentration in an unknown sample extract, the roots of the quadratic equation are solved for:

$$C_{ex} = \frac{-m_1 \pm \sqrt{(m_1)^2 - 4(m_2)(b - R_x)}}{2m_2}$$ *Equation 9*

Where:

C_{ex} = Extract analyte concentration, µg/L
R_x = Response for analyte
m_2 = Curvature
m_1 = Slope
b = y - Intercept

11.4.8 Evaluation of Second-Order Regression Calibration:

A minimum of six points must be used for a second-order regression fit.

Acceptance Criteria:

11.4.8.1 Second-order regressions should be the last option, and note that some programs (e.g., South Carolina) do not allow the use of second-order regressions.

11.4.8.2 The coefficient of determination (COD, r^2) must be ≥ 0.99.

11.4.8.3 The absolute value of the intercept is not large relative to the lowest concentrations being reported.

11.4.8.4 The response increases significantly with increasing standard concentration (i.e., the instrument response does not plateau at high concentrations).

11.4.8.5 The distribution of concentrations is adequate to characterize the curvature.

11.5 Calculations.

11.5.1 Linear Calibration Using Average Response Factors

11.5.1.1 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$ *Equation 1*

Where:

A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

11.5.1.2 For each target analyte, calculate the average response factor as follows:

$$\text{Average Response Factor} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$ *Equation 2*

Where: n = Number of calibration levels
RF_i = Response factor for the ith level

11.5.1.3 The relative standard deviation (RSD) is calculated as follows:

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\% \qquad \textit{Equation 3}$$

Where:

$\overline{RF}$ = Mean of RFs from the initial calibration for a compound
SD = Standard deviation for the mean RF from the initial calibration for a compound

$$\mathrm{SD} = \sqrt{\frac{\sum_{i=1}^{N}\left(RF_i - \overline{RF}\right)^2}{N-1}} \qquad \textit{Equation 4}$$

RF_i = RF for each of the calibration levels
n = Number of RF values

11.5.1.4 Average Calibration Fit Evaluation

The calibration relationship can be graphically represented as a line through the origin with a slope equal to the average response factor.

Acceptance Criteria: The RSD of the average response factor must be ≤15%. Also examine the residuals, especially for the high points versus the fitted function. If the residual values are large, a linear regression should be considered.

Corrective Action: **If the RSD is > 15%, average response factor cannot be used and least-squares linear regression should be attempted.**

11.5.1.5 Calculating the Concentration in the Extract

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}} \qquad \textit{Equation 5}$$

Where:

C_{ex} = Concentration in the extract, µg/mL
R_x = Response for the analyte
R_{is} = Response for the internal standard
C_{is} = Concentration of the internal standard
$\overline{RF}$ = Average response factor

11.6 Acceptance Criteria Independent of Calibration Model – Method 8000D

11.6.1 Method 8000D in SW-846 recommends the use of either the Percent Error or Relative Standard Error to evaluate the calibration model as described below.

NOTE: For South Carolina work, one of these methods of evaluation must be used.

11.6.2 Percent Error

11.6.2.1 The percent error assesses the refitting of the calibration data back to the model. The percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards except for the lowest data point which should be ≤ 50%.

11.6.2.2 Calculation of the Percent Error

$$\%\,Error = \frac{x_i - x_i'}{x_i} \times 100$$ *Equation 10*

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x'_i = True amount of analyte at calibration level i, in mass or concentration units

11.6.3 Relative Standard Error (RSE)

11.6.3.1 Relative standard error provides a measure of the fit of the calibration that is independent of the calibration model. RSE is calculated using the following equation:

$$\mathrm{RSE} = 100 \times \sqrt{\frac{\sum_{i=1}^{N}\left(\frac{x'_i - x_i}{x_i}\right)^2}{(n-p)}}$$ *Equation 11*

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units

p = Number of terms in the fitting equation
(average = 1, linear = 2, quadratic = 3, cubic = 4)

n = Number of calibration terms

11.6.3.2 The RSE acceptance limit criterion is the same as the RSD limit.

11.7 Calculations for reporting original samples

11.7.1 Calculating concentration in aqueous samples:

$$\text{Concentration, } \mu\text{g/L} = C_{ex} D$$ *Equation 12*

Where:

C_{ex}	=	Analyte concentration, µg/L
D	=	Dilution factor

11.7.2 Calculating concentration in low level soil samples:

$$\text{Concentration, } \mu\text{g/Kg} = C_{ex} \times \frac{Vp}{Ws} \times \frac{1}{(100 - M)} \times D$$ *Equation 13*

Where:

C_{ex}	=	Analyte concentration, µg/L
Vp	=	Purge volume (5 mL)
Ws	=	sample weight (in grams)
M	=	Percent Moisture
D	=	Dilution factor

Note: Units conversions for purge volume and sample weight yield conversion factor of 1 and are not shown in the equation for simplicity.

11.7.3 Calculating concentration in medium level soil samples

$$Concentration, \mu g / Kg = C_{ex} \times \frac{V_p}{V_a} \times \frac{V_e}{W_s} \times \frac{1}{(100 - M)} \times DF$$ *Equation 14*

C_{ex}	=	Uncorrected concentration of sample from instrument quantitation report (µg/L)
V_p	=	Final purge volume (mL)
V_a	=	Volume of methanol extract added to the purge volume (mL)
V_e	=	Volume of methanol used for extraction (mL)
W_s	=	Weight of sample extracted (g)
DF	=	Dilution Factor

Note: Units conversions for purge volume and sample weight yield conversion factor of 1 and are not shown in the equation for simplicity.

11.7.4 LCS and CCV Percent Recovery

$$\text{Control Spike Recovery} = \frac{S_{SR}}{S_A} \times 100\%$$ *Equation 15*

Where (in µg/L):

S_{SR}	=	Calculated analyte concentration of spiked sample
S_A	=	Concentration of standard added

11.7.5 MS/MSD Recovery

$$\%\ \text{Recovery} = \frac{SSR - SR}{SA} \times 100\%$$ *Equation 16*

Where:

SSR	=	*Spike sample result.*
SR	=	*Sample result.*

SA = *Spike added.*

11.7.6 RPD calculation for the MS/MSD:

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100\%$$ *Equation 17*

Where:

RPD = *Relative percent difference.*
MSR = *Matrix spike result.*
MSDR = *Matrix spike duplicate result.*

11.7.7 See SOP CA-Q-S-005 for more detailed calibration equations. All data are subject to two levels of technical review, documented on a checklist, as described in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

12.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in TestAmerica Denver's Policy No. CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements (e.g., DoD) indicate a greater frequency.

12.1.2 MDLV and LOQV

12.1.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes at the programmatically required spike amounts.

12.1.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

12.1.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

12.2 Demonstration of Capabilities

12.2.1 All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going

proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.2.1.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to the LCS.

12.2.1.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.2.1.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.2.1.4 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.3 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

14.2 The following waste streams are produced when this method is carried out:

14.2.1 Methanol Waste - Vial Waste and Flammable – Waste Streams A and C

14.2.2 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

14.2.3 Acidified Water – Waste Stream W

NOTE: Radioactive waste, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of these materials.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 8260B/C/D, Volatile Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 2, December, 1996.

15.1.2 Method 5030B, Purge-and-Trap for Aqueous Samples, Revision 2, December, 1996.

15.1.3 Method 5035, Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Revision 0, December, 1996.

15.1.4 Method 5035A (R1-MIR), Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Draft Revision 1, July 2002.

15.1.5 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

15.1.6 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

15.1.7 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

15.2 40 CFR Part 136, Appendix A (Method 624/624.1, Method 603) and update on August 28, 2018.

15.3 Method AK101 For the Determination of Gasoline Range Organics, Alaska DEC, Version 04/08/02.

16.0 Method Modifications:

Item	Method	Modification
1	SW-846 8260B/8260C	Ion 119 is used as the quantitation ion for chlorobenzene-d_5.
2	SW-846 8260B/8260C	The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.
3	SW-846 8260B/8260C	This SOP has been written to allow for a 20 mL purge volume for waters. An additional 5 mL of DI water is added to all samples, QC and calibration standards. The final purge volume is 25 mL.
4	SW-846 8260B/8260C	Method 8260B recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable

Item	Method	Modification
		vessels are used one time only.
5	EPA 624/624.1	Method 624/624.1 is required for demonstration of compliance with CWA permits, e.g., NPDES wastewater discharge permits. This method can be applied only to aqueous matrices. The standard analyte list and reporting limits are listed in Table A-1. If compounds are added to the analysis, all of the method criteria must be satisfied for the additional compounds.
6	EPA 624/624.1	The tune period for this method is defined as 12 hours, which is the maximum elapsed time before the tune check is performed. Calibration verifications are done at the same 12 hour frequency.
7	EPA 624/624.1	The initial calibration curve for this method requires at least five points, as shown in Table 5A.
8	EPA 624/624.1	Sample concentrations are calculated using the average RRF from the initial calibration curve.
9	EPA 624/624.1	Each target analyte is assigned to the closest eluting internal standard.
10	EPA 624/624.1	Initial demonstration of Proficiency • The spiking level for the four replicate initial demonstration of proficiency is 20 µg/L. The acceptance criteria are listed in Table A-2
11	EPA 624/624.1	Initial calibration curve requirements: • Target compounds must have RSD ≤ 35%. • If this requirement can not be met, a regression curve must be constructed for the non-compliant compounds. Then the r^2 was be greater than 0.920.
12	EPA 624/624.1	Continuing calibration verification requirements: • The continuing calibration standard is from a different source than the initial calibration standard. The daily CCAL concentration is 20 µg/L. The acceptance criteria are listed in Table A-2. Matrix Spike and LCS Requirements • The matrix spike and LCS are spiked at 20 µg/L, prepared from the same source containing all analytes of interest. A matrix spike duplicate is not necessary for this method. The recovery limits for matrix spike and LCS recovery are listed in Table A-2.
13	EPA 624/624.1	Consistent with the other volatile methods, corrections for recovery are not allowed.
14	EPA 624/624.1	Qualitative Identification – The source method states that the relative intensities of ions should agree to within ±20% between the standard and sample spectra. This SOP uses ±30%. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)
15	EPA	Section 5.2.2 of the source method describes the trap packing materials

Item	Method	Modification
	624/624.1	as Tenax GC, Methyl silicone, silica gel and coconut charcoal. TestAmerica routinely employs the OI #10 trap which consists of Tenax/Silica Gel/ Carbon Molecular Sieve or the Supelco Vocarb 3000 which consists of Carbopack B, Carbonxen1000 and 1001.
16	EPA 624/624.1	Section 5.3.2 of the source method describes a packed analytical column. TestAmerica routinely employs capillary columns when performing this method.
17	EPA 624/624.1	The source method provides a suggested list of compounds for internal and surrogate standards. Others are permitted by the method. TestAmerica uses three internal standards, including chlorobenzene-d_5 and 1,4-dichlorobenzene-d_4, which are not listed in Table 3 of the source method. Toluene-d_8 is used as a surrogate compound, which is also not listed in the source method.
18	EPA 624/624.1	The lab is preparing internal standards at 10 µg/L and applying the same criteria designed for 30 µg/L in the Method. The lower concentration is consistent with the greater sensitivity provided by capillary columns as compared to the older packed columns described in the method. It could only be more challenging for the lab to meet the acceptance criteria at 10 µg/L; it provides a higher level of data quality.
19	EPA 624/624.1	Method 624/624.1 describes a mass scan range of 25 to 260 amu. Table 13 lists all of the ions used for analysis. None of the ions are below 35 amu. Therefore, the laboratory scans from 35 to 300 and includes all ions needed for analysis.
20	EPA 624/624.1	Method 624/624.1 describes dilutions "if response of any m/z" exceeds the response for the highest m/z in the ICAL. As the m/z ratio is always directly proportional to the concentration, evaluation based on dilution (per 11.10) is equivalent.
21	EPA 624/624.1	Method 624/624.1 has criteria for unresolved isomers. The problems of isomeric resolution for the routine analytes listed in this SOP were worked through when the laboratory developed its implementation of the method. For example, we know through experience that meta- and para-xylenes will not be resolved and it was not necessary to include an evaluation for the xylenes in each analysis. meta- and para-xylenes are reported as an isomeric pair. Any development work to add compounds would take this into account.
22	624/624.1	The source method recommends Method 603 as the preferred method for Acrolein and Acrylonitrile. Method 624/624.1 is recommended as a screening method (see section 1.2 of Methods 603 and 624/624.1). Calibration and quality control samples indicate that the conditions described in this SOP are suitable for the analysis of Acrolein and Acrylonitrile. EPA's Method Update Rule (MUR), May 18, 2012, allows the addition of acrolein and acrylonitrile to Method 624/624.1, using the preservation, holding time and QC acceptance criteria from Method 603. As states implement the MUR Method 624/624.1 becomes a determinative method for these two analytes. Until such time, Method 624/624.1 remains a screening method for regulatory compliance.
23	SW846 5035	The source method recommends adding approximately the same amount of the sodium bisulfate preservative as the sample (e.g., ~ 1 g), as the

Item	Method	Modification
		presence of the preservative will affect the purging efficiencies of the analytes. TestAmerica Denver does not recommend the use of sodium bisulfate to preserve soil samples, but encourages clients to collect samples using other available methods. The use of this preservative has been shown to cause difficulties recovering more reactive analytes on the purge and trap system (e.g. 2-Chloroethyl vinyl ether, acrylamide).

17.0 Attachments

Table 1. TestAmerica Primary List Reporting Limits for 8260B/C/D
Table 2. TestAmerica 8260 Secondary List Reporting Limits
Table 3. TestAmerica Appendix IX List Reporting Limits
Table 4. TestAmerica Non-Standard Compound List Reporting Limits
Table 5. Soil Calibration Levels, 5-gram Purge (µg/Kg)
(Standard Mixes: MV-MegaMainA and MV-Gas A)
Table 5A. Soil Calibration Levels, 5 gram Purge (µg/Kg)
(Standards: MV-Supp A and Freon_A)
Table 6. Water 8260 List Calibration Levels (µg/L)
(Standards: MV-MegaMainA and MV-Gas A)
Table 6A Water 8260 List Calibration Levels (µg/L)
(Standards: MV-Supp A and Freon_A)
Table 7. Medium Level Soil 8260 List Calibration Levels (µg/Kg)
(Standards: MV-MegaMainA and MV-Gas A)
Table 7A. Medium Level Soil 8260 List Calibration Levels (µg/Kg)
(Standards: MV-Supp A and Freon_A)
Table8. Manually added Internal Standards
Table 8A. Automatically Added Internal Standards
Table 9. Manually Added Surrogate Standards
Table 9A. Automatically Added Surrogate Standards
Table 10. BFB Key Ion Abundance Criteria
Table 11. SPCC Compounds and Minimum Response Factors
Table 12. CCC Compounds
Table 13. Characteristic Ions
Table 14. State of Arizona ICV/CCV Quality Control Limits
Table 15. List 1 Poorly Performing Compounds
Table A-1. Method 624/624.1 Analytes and Reporting Limits, 5-mL Purge
Table A-2. Method 624/624.1 QC Acceptance Criteria
Table 5A. Calibration Levels for 624/624.1/624/624.1.1, 5 mL Purge
Table 17. Associated Surrogates and Internal Standards for 8260B/C/D
Appendix A Modifications for Analysis of Soils Collected for the State of Alaska
Table Ap-1: TestAmerica 8260 Reporting Limits – AK Soils
Table Ap-2: Calibration Levels for 8260, 5035FM_AK
Table Ap-3: 5035FM_AK Calibration Levels (µg/Kg)
(Standards: MV-Supp Std and MV-2 Cleve)
Attachment 1. Gas Standards Tracking Log

18.0 Changes from Previous Revision

- Revision 26, dated 13 March 2020
  - Revised correlation coefficient requirements in sections 10.7.13.12 and 11.4.6.5 to comply with 8000D requirements.

- Revision 25, dated 31 August 2019
  - Changed analysis procedures throughout SOP to reflect the changes in instrumentation from Archon auto samplers to Centurion sampling systems
  - Added procedure for scanning in documents during the data review process. See section 11.4.4
  - Updated changes to the calibration of surrogates and ICIS. See section 10.7.13.7

- Revision 24, dated 15 February 2019
  - SOP references updated to included 8260D.
- Revision 23, dated 08 January 2019
  - Updated 624 to 624/624.1
- Revision 22, dated 05 November 2018
  - Updated section 8.1.1.1 concerning headspace/bubbles.
- Revision 21, dated 30 September 2018
  - Added section 8.1.1.1 regarding headspace/bubble check requirements by Sample Receiving and Analysts.
- Revision 20, dated 31 August 2018
  - Updated for 8260C.
- Revision 19, dated 31 March 2018
  - Updated for 624/624.1/624/624.1.1.1 MUR update.
- Revision 18, dated 15 August 2017
  - Changed concentration of Ethanol in calibration tables to reflect new standard concentration.
  - Fixed spelling of Tetrachloroethene in IS/Surr association table.
  - Added instructions on handling status change of soil prep batches.
- Revision 17, dated 20 October 2016
  - Added definition for LLOQ as new Section 3.7 per 8000D
  - Removed comment about Colorado allowing 14 day holding time for unpreserved samples in existing section 8.1.3
  - Revised Holding time table for water to reflect Update V and clarify HT for method 624/624.1 based on 40 CFR Part 136; revised footnotes to the table.
  - Revised section 10.3 regarding how soil sample weights are documented to reflect current practice
  - Added note in Section 10.4.1 to NOT affix labels on terracore vials before sample is analyzed or prepped.
  - Added information on Chrom setup for TICs in new section 11.3.2
  - Added new Section 11.5 for Accetpance Criteria Independent of Calibration Model for 8000D (Percent Error and Relative Standard Error)
  - Added equations for calculation of soil concentrations in new Sections 11.6.5 and 11.6.6
  - Added MDLV and LOQV information in Section 12.1.2
  - Added reference to Method 8000D in Section 15

- Revision 16, dated 21 April 2016

- Added requirements for glassware cleaning in appropriate sections throughout.
- Added requirements for LCS and MS spikes to Sections 7.12, 7.13, 9.6 and 9.7, clarifying requirements for DoD, South Carolina and NELAP (TNI).
- Added requirement to Section 9.6 to add antifoam to LCS when added to samples.
- Clarified Section 10.7.4.4.
- Removed requirement to document use of antifoam in NCM in Section 10.7.4.12. Record made on run log.
- Revised Section 10.7.13.12 to include South Carolina criteria for ICV.
- Revised Section 10.7.14.4 to include South Carolina criteria for CCV (non-CCC compounds).
- Added new section 11.4 to list common data review findings and appropriate fixes.
- Revised Section 11.5.1.4 (old section 11.4.1.4) to correctly refer to average response factor rather than calibration factor as this is an internal standard calibration technique.
- Removed Table 10 (LCS short list) and made reference to spikes actually used throughout. Renumbered remaining tables.
- Removed references to AFCEE throughout; laboratory follows requirements of DoD QSM for all DoD work.
- Corrected terminology for evaluation of calibration using %RSD from calibration factor (external standard) to response factor (internal standard)
- Reviewed SOP to ensure that all DoD QSM 5.0 requirements were appropriately stated in addition to Section 10.10. Added specifications as needed throughout document for clarity.
- Deleted Item 5 from previous Method Modifications table. No longer applicable. Renumbered remaining items.

- Revision 15, dated 30 September 2015
  - Revised section 6.3.7 for use of disposable wooden spatulas
  - Revised Sections 9.3 and 9.5 to describe matrix used for QC samples

- Revision 14, dated 08 September 2015
  - Revised description of method blank with respect to blank matrix used for each type of analysis (Section 9.3)
  - Added description of LCS for medium level soil.
  - Revised corrective actions for matrix spike/matrix spike duplicate in Section 9.6.
  - Added notes to Section 10.7.2.1 and Appendix A requiring 12-hour tune for Method 624/624.1 for South Carolina samples.
  - Revised Section 10.10 to clarify QSM requirements and lab requested variances.
  - Updated SOP reference in Section 11.1 to current corporate SOP.
  - Added statement to Section 12.1 that MDLV be performed using matrix for method blank.
  - Added note to Method Modification #7 limiting Method 624/624.1 tune time to 12 hours for South Carolina samples.

- Revision 13, dated 30 April 2015
  - Revised discussion and preservation/HT table for soil samples in Section 8
  - Corrected retention time window in 11.2.1.1 for qualitative identification
  - Added clarification to ICAL tables in Attachments regarding use of calibration points below standard reporting limits
  - Corrected RL in water for 2-chloroethylvinyl ether
  - Corrected Table 13 entry for methyl-*tert*-butyl ether characteristic ions

- Corrected footnotes on Table A-2, Method 624/624.1 QC Acceptance Criteria to reflect current practice

- Revision 12, dated 31 December 2014
  - Added Section 10.7.14.9 to describe run sequence.

- Revision 11, dated 31 January 2014
  - Changed calibration tables to reflect changes due to standard standards
  - Added internal standard reference table (Table 17)
  - Added TBA-d_9 and 1,4-Dioxane-d_8 as internal standards
  - Removed SIM (see SOP DV-MS-0016)
  - Removed section 9.8

- Revision 10, dated 19 August 2013
  - Formatting changes

- Revision 9, dated 04 January 2013
  - Added section 9.8 to address the 2012 MUR QC requirements

- Revision 8, dated 28 September 2012
  - Added to compounds to the reporting limit, characteristic ion and calibration tables to match TALS.

- Revision 7, dated 27 July 2012
  - Added sodium bisulfate to Section 7.
  - Revised Section 8 to include Terra Core samplers and moved instructions on sample preparation and handling in the lab to Section 10. Reorganized sampling and preservation information into tables. Updated information including footnote on Holding Time and preservation table for water regarding Method Update Rule that approves use of Method 624/624.1 for analysis of acrolein and acrylonitrile.
  - Removed flowcharts from Section 8.
  - Revised Section 9.1
  - Revised Section 10.
  - Updated reference section to include Method 603, Method 5035A, and Method 8000B and 8000C.
  - Revised Method Modifications #23
  - Updated tables to reflect current practice.
  - Added Appendix A for the analysis of soils using the AK methanol extraction procedure.
  - Formatting and editorial changes throughout

- Revision 6.4, dated 28 December 2011
  - Changed the column ID and film thickness in section 6.1.8.1

- Revision 6.3, dated 26 October 2011
  - Added Section 4.6 regarding interferences with toluene-d_8 surrogate when potassium permanganate may have been added to sample
  - Updated path to QAS folders in the public folders, section 9.7
  - Added J. T. Baker Antifoam B and reagent sand, sections 7.3, 7.4
  - Added description of procedure for use of antifoaming agent B, section 10.1.3.8
  - Formatting

- Revision 6.2, dated 25 August, 2011
  - Added requirements to section 9.4 for the use of Ottawa sand in soil LCS's.

- Revision 6.1, dated 31 January, 2011
  - Added Attachment 1, Gas Standards Tracking Log
  - Added section 11.1 referencing corporate SOP CA-Q-S-005 "Calibration Curves"

Earlier revision histories have been archived and are available upon request.

Table 1. TestAmerica Primary List Reporting Limits for 8260B/C/D

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
Dichlorodifluoromethane	75-71-8	2	10	500
Chloromethane	74-87-3	2	10	500
Bromomethane	74-83-9	2	10	500
Vinyl chloride	75-01-4	1	5	500
Chloroethane	75-00-3	2	10	500
Trichlorofluoromethane	75-69-4	2	10	500
Acrolein	107-02-8	20	50	5,000
Acetone	67-64-1	10	20	1,000
Trichlorotrifluoroethane	76-13-1	3	20	1,000
Ethanol	64-17-5	300	600	10,000
Iodomethane	74-88-4	1	5	250
Carbon disulfide	75-15-0	2	5	250
Methylene chloride	75-09-2	2	5	250
tert-Butyl alcohol	75-65-0	50	200	10,000
1,1-Dichloroethene	75-35-4	1	5	250
1,1-Dichloroethane	75-34-3	1	5	250
trans-1,2-Dichloroethene	156-60-5	1	2.5	125
Acrylonitrile	107-13-1	20	50	5,000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	5	20	250
Hexane	110-54-3	2	5	250
cis-1,2-Dichloroethene	156-59-2	1	2.5	125
1,2-Dichloroethene (Total)	540-59-0	1	5	250
Tetrahydrofuran	109-99-9	7	20	1,000
Chloroform	67-66-3	1	10	250
1,2-Dichloroethane	107-06-2	1	5	250
Dibromomethane	74-95-3	1	5	250
2-Butanone	78-93-3	6	20	1,000
1,4-Dioxane	123-91-1	200	500	25,000
1,1,1-Trichloroethane	71-55-6	1	5	250
Carbon tetrachloride	56-23-5	1	5	250
Bromodichloromethane	75-27-4	1	5	250
1,2-Dichloropropane	78-87-5	1	5	250

Table 1. TestAmerica Primary List Reporting Limits for 8260B/C/D (continued)

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
cis-1,3-Dichloropropene	10061-01-5	1	5	250
Trichloroethene	79-01-6	1	5	250
Dibromochloromethane	124-48-1	1	5	250
1,2-Dibromoethane	106-93-4	1	5	250
1,2,3-Trichloropropane	96-18-4	2.5	5	250
1,1,2-Trichloroethane	79-00-5	1	5	250
Benzene	71-43-2	1	5	250
Ethylmethacrylate	97-63-2	3	5	250
trans-1,3-Dichloropropene	10061-02-6	3	5	250
Bromoform	75-25-2	1	5	250
4-Methyl-2-pentanone	108-10-1	5	20	1,000
2-Hexanone	591-78-6	5	20	1,000
Tetrachloroethene	127-18-4	1	5	250
Toluene	108-88-3	1	5	250
1,1,2,2-Tetrachloroethane	79-34-5	1	5	250
2-Chloroethyl vinyl ether[2]	110-75-8	3[2]	50	2,500
Vinyl acetate	108-05-4	3	10	500
Chlorobenzene	108-90-7	1	5	250
Ethylbenzene	100-41-4	1	5	250
Styrene	100-42-5	1	5	250
trans-1,4-Dichloro-2-butene	110-57-6	3	5	250
m- and p-Xylenes	179601-23-1	2	3.5	250
o-xylene	95-47-6	1	2.5	125
Total xylenes	1330-20-7	2	10	250
1,3-Dichlorobenzene	541-73-1	1	5	250
1,4-Dichlorobenzene	106-46-7	1	5	250
1,2-Dichlorobenzene	95-50-1	1	5	250

1 Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

2 2-Chloroethylvinyl ether cannot be reliably recovered from acid preserved samples

Table 2. TestAmerica 8260 Secondary List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
2,2-Dichloropropane	590-20-7	1	5	250
Bromochloromethane	74-97-5	1	5	250
1,1-Dichloropropene	563-58-6	1	5	250
1,3-Dichloropropane	142-28-9	1	5	250
1-Chlorohexane	544-10-5	1	5	500
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
Isopropylbenzene	98-82-8	1	5	250
Bromobenzene	108-86-1	1	5	250
n-Propylbenzene	103-65-1	1	5	250
2-Chlorotoluene	95-49-8	1	5	250
4-Chlorotoluene	106-43-4	1	5	250
1,3,5-Trimethylbenzene	108-67-8	1	5	250
tert-Butylbenzene	98-06-6	1	5	250
1,2,4-Trimethylbenzene	95-63-6	1	5	250
sec-Butylbenzene	135-98-8	1	5	250
4-Isopropyltoluene	99-87-6	1	5	250
n-Butylbenzene	104-51-8	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	5	250
1,2,4-Trichlorobenzene	120-82-1	1	5	250
Naphthalene	91-20-3	1	5	500
Hexachlorobutadiene	87-68-3	1	5	250
1,2,3-Trichlorobenzene	87-61-6	1	5	250
2-Pentanone	107-87-9	5	10	500
cis-1,4-Dichloro-2-butene	1476-11-5	3	5	250
Ethylene oxide	75-21-8	600	3,000	150,000

Table 3. TestAmerica Appendix IX List Reporting Limits

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water µg/L	Low Soil µg/kg	Medium Soil µg/kg
Allyl Chloride	107-05-1	2	10	500
Acetonitrile	75-05-8	30	100	5,000
Dichlorofluoromethane	75-43-4	2	10	25,000
Isopropyl ether	108-20-3	10	50	2,500
Chloroprene	126-99-8	1	5	500
n-Butanol	71-36-3	60	200	10,000
Propionitrile	107-12-0	20	50	1,000
Methacrylonitrile	126-98-7	10	50	2,500
Isobutanol	78-83-1	110	200	10,000
Methyl methacrylate	80-62-6	4	5	250
1,1,1,2-Tetrachloroethane	630-20-6	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	5	10	500
Ethyl ether	60-29-7	2	10	500
Ethyl Acetate	141-78-6	5	10	500
2-Nitropropane	79-46-9	5	10	500
Cyclohexanone[2]	108-94-1	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	1	5	250

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.
[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4. TestAmerica Non-Standard List Reporting Limits for 8260B/C/D

Compound	CAS Number	Reporting Limits[1]		
		20 mL Water(µg/L)	Low Soil (µg/kg)	Med Soil (µg/kg)
1,1,1-Trifluoro-2,2-Dichloroethane	306.83-2	2.0	5.0	1000
1,2,3-Trimethylbenzene	526-73-8	2.0	5.0	250
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	2.0	5.0	250
1,2-Dichloro-1,1,2-Trifluoroethane	354-23-4	2.0	5.0	250
1,3,5-Trichlorobenzene	108-70-3	5.00	***	***
2,2,3-Trimethylbutane	464-06-2	5.00	***	***
2,2-Dimethylpentane	590-35-2	5.00	***	***
2,3-Dimethylpentane	565-59-3	5.00	***	***
2,4-Dimethylpentane	108-08-7	5.00	***	***
2-Chloro-1,1,1-Trifluoroetane	75-88-7	5.00	5.0	250
2-Methylhexane	591-76-4	5.00	***	***
3,3-Dimethylpentane	562-49-2	5.00	***	***
3-Ethylpentane	617-78-7	5.00	***	***
3-Methylhexane	589-34-4	5.00	***	***
Chlorotrifluoroethene	79-38-9	5.00	5.0	250
Cyclohexane	110-82-7	2.0	5.0	250
Dimethyl Disulfide	624/624.1-92-0	5.00	***	***
Isopropyl Alcohol	67-63-0	40	200	10,000
Methyl Acetate	79-20-9	5.0	10	1000
Methylcyclohexane	108-87-2	1.0	5.0	250
n-Heptane	142-82-5	5.00	***	***
n-Nonyl Aldehyde	124-19-6	10.00	***	***
Pentachloroethane[2]	76-01-7	0.5	***	***
Propene Oxide	75-56-9	50	3000	250
Sec-Butyl Alcohol	78-92-2	***	200	***
Tert-amyl methyl ether	994-05-8	5	5.0	1000
Tert-butyl ethyl ether	637-92-3	5	5.0	1000
Tetrahydrothiophene	110-01-0	2.0	5.0	***

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

[2] Due to breakdown, QC and samples being analyzed for Pentachlroethane must be preserved with acid (pH<2).

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water
(Standard Mixes: MV-MegaMainA and MV-Gas A)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	
1,1,1,2-Tetrachloroethane	0.5	1	2	10	50	100	200	
1,1,1-Trichloroethane	0.5	1	2	10	50	100	200	
1,1,2,2-Tetrachloroethane	0.5	1	2	10	50	100	200	
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.5	1	2	10	50	100	200	
1,1,2-Trichloroethane	0.5	1	2	10	50	100	200	
1,1-Dichloroethane	0.5	1	2	10	50	100	200	
1,1-Dichloroethene	0.5	1	2	10	50	100	200	
1,1-Dichloropropene	0.5	1	2	10	50	100	200	
1,2,3-Trichlorobenzene	0.5	1	2	10	50	100	200	
1,2,3-Trichloropropane	0.5	1	2	10	50	100	200	
1,2,4-Trichlorobenzene	0.5	1	2	10	50	100	200	
1,2,4-Trimethylbenzene	0.5	1	2	10	50	100	200	
1,2-Dibromo-3-chloropropane	0.5	1	2	10	50	100	200	
1,2-Dichlorobenzene	0.5	1	2	10	50	100	200	
1,2-Dichloroethane	0.5	1	2	10	50	100	200	
1,2-Dichloropropane	0.5	1	2	10	50	100	200	
1,3,5-Trimethylbenzene	0.5	1	2	10	50	100	200	
1,3-Dichlorobenzene	0.5	1	2	10	50	100	200	
1,3-Dichloropropane	0.5	1	2	10	50	100	200	
1,4-Dichlorobenzene	0.5	1	2	10	50	100	200	
1,4-Dioxane	10	20	40	200	1,000	2,000	4,000	
1-Chlorohexane	0.5	1	2	10	50	100	200	
2-Chloroethyl vinyl ether	0.5	1	2	10	50	100	200	
2,2-Dichloropropane	0.5	1	2	10	50	100	200	
2-Butanone	2	4	8	40	200	400	800	
2-Chlorotoluene	0.5	1	2	10	50	100	200	
2-Hexanone	2	4	8	40	200	400	800	
2-Pentanone	2	4	8	40	200	400	800	
3-Chloro-1-propene (Allyl Chloride	0.5	1	2	10	50	100	200	
4-Chlorotoluene		1	2	10	50	100	200	
4-Isopropyltoluene		1	2	10	50	100	200	

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water
(Standard Mixes: MV-MegaMainA and MV-Gas A)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	
4-Methyl-2-pentanone		4	8	40	200	400	800	
Acetone	2	4	8	40	200	400	800	
Acrolein	5	10	20	100	500	1,000	2,000	
Acrylonitrile	5	10	20	100	500	1,000	2,000	
Benzene	0.5	1	2	10	50	100	200	
Bromobenzene	0.5	1	2	10	50	100	200	
Bromoform	0.5	1	2	10	50	100	200	
Bromomethane	0.5	1	2	10	50	100	200	
Carbon Disulfide	0.5	1	2	10	50	100	200	
Carbon tetrachloride	0.5	1	2	10	50	100	200	
Chlorobenzene	0.5	1	2	10	50	100	200	
Chlorobromomethane	0.5	1	2	10	50	100	200	
Chloroethane	0.5	1	2	10	50	100	200	
Chloroform	0.5	1	2	10	50	100	200	
Chloromethane	0.5	1	2	10	50	100	200	
cis-1,2-Dichloroethene	0.5	1	2	10	50	100	200	
cis-1,3-Dichloropropene	0.5	1	2	10	50	100	200	
Cyclohexane	0.5	1	2	10	50	100	200	
Cyclohexanone	20	40	80	400	2,000	4,000	8,000	
Chlorodibromomethane	0.5	1	2	10	50	100	200	
Dibromomethane	0.5	1	2	10	50	100	200	
Dichlorobromomethane	0.5	1	2	10	50	100	200	
Dichlorofluoromethane	0.5	1	2	10	50	100	200	
Dichlorodifluoromethane	0.5	1	2	10	50	100	200	
Ethylbenzene	0.5	1	2	10	50	100	200	
Ethyl Ether	0.5	1	2	10	50	100	200	
Ethyl methacrylate	0.5	1	2	10	50	100	200	
Ethylene dibromide	0.5	1	2	10	50	100	200	
Hexachlorobutadiene	0.5	1	2	10	50	100	200	
Hexane	0.5	1	2	10	50	100	200	
Iodomethane	0.5	1	2	10	50	100	200	
Isobutyl alcohol	12.5	25	50	250	1,250	2,500	5,000	
Isopropylbenzene	0.5	1	2	10	50	100	200	

Calibration levels:
Table 5. 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water
(Standard Mixes: MV-MegaMainA and MV-Gas A)

Compound	Calibration Level, µg/Kg							
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	
m- and p-Xylenes	0.5	1	2	10	50	100	200	
Methyl Acetate	2.5	5	10	50	250	500	1,000	
Methylcyclohexane	0.5	1	2	10	50	100	200	
Methylene chloride	0.5	1	2	10	50	100	200	
Methyl tert-butyl ether (MTBE)	0.5	1	2	10	50	100	200	
Naphthalene	0.5	1	2	10	50	100	200	
n-Butylbenzene	0.5	1	2	10	50	100	200	
n-Propylbenzene	0.5	1	2	10	50	100	200	
o-Xylene	0.5	1	2	10	50	100	200	
sec-Butylbenzene	0.5	1	2	10	50	100	200	
sec- Butyl Alcohol	15	30	60	300	1,500	3,000	6,000	
Styrene	0.5	1	2	10	50	100	200	
2-Methyl-2-propanol (tert-Butyl alcohol)	5	10	20	100	500	1,000	2,000	
tert-Butylbenzene	0.5	1	2	10	50	100	200	
Tetrachloroethene	0.5	1	2	10	50	100	200	
Tetrahydrofuran	1	2	4	20	100	200	400	
Toluene	0.5	1	2	10	50	100	200	
trans-1,2-Dichloroethene	0.5	1	2	10	50	100	200	
trans-1,3-Dichloropropene	0.5	1	2	10	50	100	200	
trans-1,4-Dichloro-2-butene	0.5	1	2	10	50	100	200	
Trichloroethene	0.5	1	2	10	50	100	200	
Trichlorofluoroethane	0.5	1	2	10	50	100	200	
Vinyl Acetate	0.5	2	4	20	100	200	400	
Vinyl chloride	0.5	1	2	10	50	100	200	

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Calibration levels:
Table 5A: 8260B/C/D Soil-5mL Purge, Aqueous-5mL purge 624/624.1[1] and 8260C/D water and soil-5mL purge
(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloro-ethane	2	5	10	20	50	100	200
1,2,3-Trimethylbenzene	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2,2-tetrafluroethane	2	5	10	20	50	100	200
1,2-Dichloro-1,1,2-trifluoroethane	2	5	10	20	50	100	200
2-Chloro-1,1,1-Trifluoroethane	2	5	10	20	50	100	200
2-Chloro-1,3-butadiene (Chloroprene)	2	5	10	20	50	100	200
2-Nitropropane	4	10	20	40	100	200	400
Acetonitrile	20	50	100	200	500	1,000	2,000
cis-1,4-Dichloro-2-butene	2	5	10	20	50	100	200
Chlorotrifluoroethene	2	5	10	20	50	100	200
Ethanol	120	300	600	1200	3000	6,000	12,000
Ethyl Acetate	4	10	20	40	100	200	400
Ethylene Oxide	400	1,000	2,000	4,000	10,000	20,000	40,000
Isopropyl alcohol	20	50	100	200	500	1,000	2,000
Isopropyl ether	2	5	10	20	50	100	200
Methylacrylonitrile	20	50	100	200	500	1,000	2,000
Methyl methacrylate	4	10	20	40	100	200	400
n-Butanol	50	125	250	500	1,250	2,500	5,000
Propene Oxide	100	250	500	1,000	2,500	5,000	10,000
Propionitrile	20	50	100	200	500	1,000	2,000
tert-Amyl methyl ether	2	5	10	20	50	100	200
tert-Butyl ethyl ether	2	5	10	20	50	100	200
Tetrahydrothiophene	2	5	10	20	50	100	200

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-MegaMain A and MV-Gas A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,1-Trichloroethane	0.5*	1.0	2.0	5.0	10	25	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.5*	1.0	2.0	5.0	10	25	60
1,1,2-Trichloroethane	0.5*	1.0	2.0	5.0	10	25	60
1,1-Dichloroethane	0.5*	1.0	2.0	5.0	10	25	60
1,1-Dichloroethene	0.5*	1.0	2.0	5.0	10	25	60
1,1-Dichloropropene	0.5*	1.0	2.0	5.0	10	25	60
1,2,3-Trichlorobenzene	0.5*	1.0	2.0	5.0	10	25	60
1,2,3-Trichloropropane	0.5*	1.0	2.0	5.0	10	25	60
1,2,4-Trichlorobenzene	0.5*	1.0	2.0	5.0	10	25	60
1,2,4-Trimethylbenzene	0.5*	1.0	2.0	5.0	10	25	60
1,2-Dibromo-3-chloropropane	0.5*	1.0	2.0	5.0	10	25	60
1,2-Dichlorobenzene	0.5*	1.0	2.0	5.0	10	25	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichloropropane	0.5*	1.0	2.0	5.0	10	25	60
1,3,5-Trimethylbenzene	0.5*	1.0	2.0	5.0	10	25	60
1,3-Dichlorobenzene	0.5*	1.0	2.0	5.0	10	25	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	25	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,4-Dioxane	10*	20	40	100	200	600	1,200
1-Chlorohexane	0.5*	1.0	2.0	5.0	10	25	60
2-Chloroethyl vinyl ether	0.5*	1.0*	2.0	5.0	10	25	60
2,2-Dichloropropane	0.5*	1.0	2.0	5.0	10	25	60
2-Butanone (MEK)	2.0*	4.0	8.0	20	40	120	240
2-Chlorotoluene	0.5*	1.0	2.0	5.0	10	25	60
2-Hexanone	2.0*	4.0	8.0	20	40	120	240
2-Pentanone	2.0*	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl Chloride)	0.5*	1.0	2.0	5.0	10	25	60
2-Methyl-2-propanol (tert-Butyl alcohol)	5.0*	10	20	50	100	250	600
4-Chlorotoluene	0.5*	1.0	2.0	5.0	10	25	60
4-Isopropyltoluene	0.5*	1.0	2.0	5.0	10	25	60
4-Methyl-2-pentanone	2.0*	4.0	8.0	20	40	120	240
Acetone	2.0*	4.0*	8.0	20	40	120	240

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-MegaMain A and MV-Gas A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Acrolein	5.0*	10	20	50	100	250	600
Acrylonitrile	5.0*	10	20	50	100	250	600
Benzene	0.5	1.0	2.0	5.0	10	25	60
Bromobenzene	0.5*	1.0	2.0	5.0	10	25	60
Bromoform	0.5*	1.0	2.0	5.0	10	25	60
Bromomethane	0.5*	1.0	2.0	5.0	10	25	60
Carbon Disulfide	0.5*	1.0*	2.0	5.0	10	25	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	25	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	25	60
Chlorobromomethane	0.5*	1.0	2.0	5.0	10	25	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	25	60
Chloroethane	0.5*	1.0*	2.0	5.0	10	25	60
Chloroform	0.5	1.0	2.0	5.0	10	25	60
Chloromethane	0.5*	1.0*	2.0	5.0	10	25	60
cis-1,2-Dichloroethene	0.5*	1.0	2.0	5.0	10	25	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	25	60
Cyclohexane	0.5*	1.0	2.0	5.0	10	25	60
Cyclohexanone	20*	40	80	200	400	1,200	2,400
Dibromomethane	0.5*	1.0	2.0	5.0	10	25	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	25	60
Dichlorofluoromethane	0.5*	1.0	2.0	5.0	10	25	60
Dichlorodifluoromethane	0.5*	1.0*	2.0	5.0	10	25	60
Ethylbenzene	0.5*	1.0	2.0	5.0	10	25	60
Ethyl Ether	0.5*	1.0	2.0	5.0	10	25	60
Ethyl methacrylate	0.5*	1.0	2.0	5.0	10	25	60
Ethylene dibromide (EDB)	0.5*	1.0	2.0	5.0	10	25	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	25	60
Hexane	0.5*	1.0	2.0	5.0	10	25	60
Iodomethane	0.5*	1.0	2.0	5.0	10	25	60
Isobutyl alcohol	12.5*	25	50	125	250	750	1,500
Isopropylbenzene	0.5*	1.0	2.0	5.0	10	25	60
m and p Xylenes	0.5*	1.0	2.0	5.0	10	25	60
Methyl Acetate	1.0*	5.0	10	25	50	150	300
Methylcyclohexane	0.5*	1.0	2.0	5.0	10	25	60
Methylene chloride	0.5*	1.0	2.0	5.0	10	25	60

Table 6: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-MegaMain A and MV-Gas A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Methyl tert-butyl ether (MTBE)	0.5*	1.0	2.0	5.0	10	25	60
Naphthalene	0.5*	1.0	2.0	5.0	10	25	60
n-Butylbenzene	0.5*	1.0	2.0	5.0	10	25	60
n-Propylbenzene	0.5*	1.0	2.0	5.0	10	25	60
o-Xylene	0.5*	1.0	2.0	5.0	10	25	60
sec-Butylbenzene	0.5*	1.0	2.0	5.0	10	25	60
sec-Butyl Alcohol	15*	30	60	150	300	900	1,800
Styrene	0.5*	1.0	2.0	5.0	10	25	60
tert-Butylbenzene	0.5*	1.0	2.0	5.0	10	25	60
Tetrachloroethene	0.5*	1.0	2.0	5.0	10	25	60
Tetrahydrofuran	0.6*	2.0	4.0	10	20	60	120
Toluene	0.5*	1.0	2.0	5.0	10	25	60
trans-1,2-Dichloroethene	0.5*	1.0	2.0	5.0	10	25	60
trans-1,3-Dichloropropene	0.5*	1.0	2.0	5.0	10	25	60
trans-1,4-Dichloro-2-butene	0.5*	1.0	2.0	5.0	10	25	60
Trichloroethene	0.5*	1.0	2.0	5.0	10	25	60
Trichlorofluoromethane	0.5*	1.0*	2.0	5.0	10	25	60
Vinyl Acetate	1.0*	2.0	4.0	10	20	60	120
Vinyl chloride	0.5	1.0	2.0	5.0	10	25	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
* These compounds are not routinely used at this concentration of the calibration.
NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Table 6A: Water 8260B/C/D-20mL purge List Calibration Levels (µg/L)[1]
(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
1,1,1-Trifluoro-2,2-dichloroethane	1.0	2.0	5.0	10	25	60
1,2,3-Trimethylbenzene	1.0	2.0	5.0	10	25	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	1.0	2.0	5.0	10	25	60
1,2-Dichloro-1,1,2-trifluoroethane	1.0	2.0	5.0	10	25	60
2-Chloro-1,1,1-Trifluoroethane	1.0	2.0	5.0	10	25	60
2-Chloro-1,3-butadiene (Chloroprene)	1.0	2.0	5.0	10	25	60
2-Nitropropane	1.0	2.0	5.0	10	25	60
Acetonitrile	10	20	50	100	250	600
cis-1,4-dichloro-2-butene	1.0	2.0	5.0	10	25	60
Chlorotrifluoroethene	1.0	2.0	5.0	10	25	60
Ethanol	60	120	300	600	1,800	3,600
Ethyl acetate	2.0	4.0	10	20	60	120
Ethylene oxide	200	400	1,000	2,000	6,000	12,000
Isopropyl Alcohol	10	20	50	100	250	600
Isopropyl Ether	1.0	2.0	5.0	10	25	60
Methylacrylonitrile	10	20	50	100	250	600
Methyl methacrylate	2.0	4.0	8.0	20	60	120
n-Butanol	25	50	125	250	750	1,000
Propene oxide	50	100	250	500	1,500	3,000
Propionitrile	10	20	50	100	250	600
tert-Amyl methyl ether	1.0	2.0	5.0	10	25	60
tert-Butyl ethyl ether	1.0	2.0	5.0	10	25	60
Tetrahydrothiophene	1.0	2.0	5.0	10	25	60
1,2-Dichloroethane-d4 (surr)	1.0*	2.0	5.0	10	25	60
Toluene-d8 (surr)	1.0*	2.0	5.0	10	25	60
4-Bromofluorobenzene (surr)	1.0*	2.0	5.0	10	25	60
Dibromofluoromethane (surr)	1.0*	2.0	5.0	10	25	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
* These compounds are not routinely used at this concentration of the calibration.
NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-MegaMain A and MV-Gas A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,1-Trichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,2,2-Tetrachloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.5	1.0	2.0	5.0	10	25	60
1,1,2-Trichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1-Dichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,1-Dichloroethene	0.5	1.0	2.0	5.0	10	25	60
1,1-Dichloropropene	0.5	1.0	2.0	5.0	10	25	60
1,2,3-Trichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,2,3-Trichloropropane	0.5	1.0	2.0	5.0	10	25	60
1,2,4-Trichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,2,4-Trimethylbenzene	0.5	1.0	2.0	5.0	10	25	60
1,2-Dibromo-3-chloropropane	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichloropropane	0.5	1.0	2.0	5.0	10	25	60
1,3,5-Trimethylbenzene	0.5	1.0	2.0	5.0	10	25	60
1,3-Dichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,3-Dichloropropane	0.5	1.0	2.0	5.0	10	25	60
1,4-Dichlorobenzene	0.5	1.0	2.0	5.0	10	25	60
1,4-Dioxane	10	20	40	100	200	600	1,200
1-Chlorohexane	0.5	1.0	2.0	5.0	10	25	60
2-Chloroethyl vinyl ether	0.5	1.0	2.0	5.0	10	25	60
2,2-Dichloropropane	0.5	1.0	2.0	5.0	10	25	60
2-Butanone (MEK)	2.0	4.0	8.0	20	40	120	240
2-Chlorotoluene	0.5	1.0	2.0	5.0	10	25	60
2-Hexanone	2.0	4.0	8.0	20	40	120	240
2-Pentanone	2.0	4.0	8.0	20	40	120	240
3-Chloro-1-propene (Allyl Chloride)	0.5	1.0	2.0	5.0	10	25	60
2-Methyl-2-propanol (tert-Butyl alcohol)	10	20	40	100	200	600	1,200
4-Chlorotoluene	0.5	1.0	2.0	5.0	10	25	60
4-Isopropyltoluene	0.5	1.0	2.0	5.0	10	25	60
4-Methyl-2-pentanone	2.0	4.0	8.0	20	40	120	240
Acetone	2.0	4.0	8.0	20	40	120	240
Acrolein	5	10	20	50	100	250	600

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-MegaMain A and MV-Gas A) (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Acrylonitrile	5	10	20	50	100	250	600
Benzene	0.5	1.0	2.0	5.0	10	25	60
Bromobenzene	0.5	1.0	2.0	5.0	10	25	60
Bromoform	0.5	1.0	2.0	5.0	10	25	60
Bromomethane	0.5	1.0	2.0	5.0	10	25	60
Carbon Disulfide	0.5	1.0	2.0	5.0	10	25	60
Carbon tetrachloride	0.5	1.0	2.0	5.0	10	25	60
Chlorobenzene	0.5	1.0	2.0	5.0	10	25	60
Chlorobromomethane	0.5	1.0	2.0	5.0	10	25	60
Chlorodibromomethane	0.5	1.0	2.0	5.0	10	25	60
Chloroethane	0.5	1.0	2.0	5.0	10	25	60
Chloroform	0.5	1.0	2.0	5.0	10	25	60
Chloromethane	0.5	1.0	2.0	5.0	10	25	60
cis-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	25	60
cis-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	25	60
Cyclohexane	0.5	1.0	2.0	5.0	10	25	60
Cyclohexanone	20	40	80	200	400	1,200	2,400
Dibromomethane	0.5	1.0	2.0	5.0	10	25	60
Dichlorobromomethane	0.5	1.0	2.0	5.0	10	25	60
Dichlorofluoromethane	0.5	1.0	2.0	5.0	10	25	60
Dichlorodifluoromethane	0.5	1.0	2.0	5.0	10	25	60
Ethylbenzene	0.5	1.0	2.0	5.0	10	25	60
Ethyl Ether	0.5	1.0	2.0	5.0	10	25	60
Ethyl Methacrylate	0.5	1.0	2.0	5.0	10	25	60
Ethylene dibromide (EDB)	0.5	1.0	2.0	5.0	10	25	60
Hexachlorobutadiene	0.5	1.0	2.0	5.0	10	25	60
Hexane	0.5	1.0	2.0	5.0	10	25	60
Iodomethane	0.5	1.0	2.0	5.0	10	25	60
Isobutyl Alcohol	12.5	25	50	125	250	750	1,500
Isopropylbenzene	0.5	1.0	2.0	5.0	10	25	60
m and p Xylenes	0.5	1.0	2.0	5.0	10	25	60
Methyl Acetate	2.5	5.0	10	25	50	150	300
Methylcyclohexane	0.5	1.0	2.0	5.0	10	25	60
Methylene chloride	0.5	1.0	2.0	5.0	10	25	60
Methy tert-butyl ether	0.5	1.0	2.0	5.0	10	25	60

Table 7: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-MegaMain A and MV-Gas A) (cont.)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Naphthalene	0.5	1.0	2.0	5.0	10	25	60
n-Butylbenzene	0.5	1.0	2.0	5.0	10	25	60
n-Propylbenzene	0.5	1.0	2.0	5.0	10	25	60
o-Xylene	0.5	1.0	2.0	5.0	10	25	60
sec-Butylbenzene	0.5	1.0	2.0	5.0	10	25	60
sec-Butyl Alcohol	15	30	60	150	300	900	1,800
Styrene	0.5	1.0	2.0	5.0	10	25	60
tert-Butylbenzene	0.5	1.0	2.0	5.0	10	25	60
Tetrachloroethene	0.5	1.0	2.0	5.0	10	25	60
Tetrahydrofuran	1.0	2.0	4.0	10	20	60	120
Toluene	0.5	1.0	2.0	5.0	10	25	60
trans-1,2-Dichloroethene	0.5	1.0	2.0	5.0	10	25	60
trans-1,3-Dichloropropene	0.5	1.0	2.0	5.0	10	25	60
trans-1,4-Dichloro-2-butene	0.5	1.0	2.0	5.0	10	25	60
Trichloroethene	0.5	1.0	2.0	5.0	10	25	60
Trichlorofluoromethane	0.5	1.0	2.0	5.0	10	25	60
Vinyl Acetate	1.0	2.0	4.0	10	20	60	120
Vinyl chloride	0.5	1.0	2.0	5.0	10	25	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Table 7A: Medium Level Soil 8260B/C/D List Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp A and Freon_A)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
1,1,1-Trifluoro-2,2-dichloroethane	0.5	1.0	2.0	5.0	10	25	60
1,2,3-Trimethylbenzene	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichloro-1,1,2,2-tetrafluoroethane	0.5	1.0	2.0	5.0	10	25	60
1,2-Dichloro-1,1,2-trifluoroethane	0.5	1.0	2.0	5.0	10	25	60
2-Chloro-1,1,1-Trifluoroethane	0.5	1.0	2.0	5.0	10	25	60
2-Chloro-1,3-butadiene (Chloroprene)	0.5	1.0	2.0	5.0	10	25	60
2-Nitropropane	0.5	1.0	2.0	5.0	10	25	60
Acetonitrile	5.0	10	20	50	100	250	600
cis-1,4-dichloro-2-butene	0.5	1.0	2.0	5.0	10	25	60
Chlorotrifluoroethene	0.5	1.0	2.0	5.0	10	25	60
Ethanol	30	60	120	300	600	1,800	3,600
Ethyl acetate	1.0	2.0	4.0	10	20	60	120
Ethylene oxide	100	200	400	1,000	2,000	6,000	12,000
Isopropyl Alcohol	5	10	20	50	100	250	600
Isopropyl Ether	0.5	1.0	2.0	5.0	10	25	60
Methylacrylonitrile	5	10	20	50	100	250	600
Methyl methacrylate	1.0	2.0	4.0	8.0	20	60	120
n-Butanol	12.5	25	50	125	250	750	1,000
Propene oxide	25	50	100	250	500	1,500	3,000
Propionitrile	5	10	20	50	100	250	600
tert-Amyl methyl ether	0.5	1.0	2.0	5.0	10	25	60
tert-Butyl ethyl ether	0.5	1.0	2.0	5.0	10	25	60
Tetrahydrothiophene	0.5	1.0	2.0	5.0	10	25	60

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.
NOTE: The lab has the option to either calibrate the surrogates at the same concentrations as the other analytes or to run at least 5 replicates of the surrogates at the same concentration.

Table 8. Manually Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	20	96
Chlorobenzene-d_5	20	119
1,4-Dichlorobenzene-d_4	20	152
TBA-d9	400	65
1,4-Dioxane-d8*	400	96

NOTES:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.

Table 8A. Automatically Added Internal Standards

Internal Standard	Standard Concentration (µg/mL)	Quantitation Ion
Fluorobenzene	250	96
Chlorobenzene-d_5	250	119
1,4-Dichlorobenzene-d_4	250	152
TBA-d9	5000	65
1,4-Dioxane-d8*	5000	96

NOTES:

1) 1 µL of the internal standard is added to the sample. This results in a concentration of each internal standard in the sample at 10 µg/L for a 20 mL purge.

2) There may be some variability in the size of the internal standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added internal standard concentrations.

* 1,4-Dioxane-d8 is in the Internal Standard spiked into every sample but is not associated to any analytes due to poor stability. 1,4-Dioxane must be set to “rejected” or “not needed” status in TALs.

Table 9. Manually Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	20
Dibromofluoromethane	20
Toluene-d_8	20
4-Bromofluorobenzene	20

NOTES:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.
2) Except for high-level soils, the surrogate and internal standards may be combined in one solution.
3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9A. Automatically Added Surrogate Standards

Surrogate Compounds	Standard Concentration (µg/mL)
1,2-Dichloroethane-d_4	250
Dibromofluoromethane	250
Toluene-d_8	250
4-Bromofluorobenzene	250

NOTES:

1) 1 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample at 10 µg/L for a 20 mL purge.
2) There may be some variability in the size of the surrogate standard loop from one instrument to the next. This is compensated for on the day of initial calibration by comparing the manually added and automatically added surrogate standard concentrations.
3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 10. BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15 to 40 % of Mass 95
75	30 to 60 % of Mass 95
95	Base Peak, 100 % Relative Abundance
96	5 to 9 % of Mass 95
173	Less than 2 % of Mass 174
174	Greater than 50 % of Mass 95
175	5 to 9 % of Mass 174
176	Greater than 95 %, but less than 101 % of Mass 174
177	5 to 9 % of Mass 176

Table 11. SPCC Compounds and Minimum Response Factors

Compound	8260B Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	> 0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

Table 12. CCC Compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	≤ 30.0	≤ 20.0
1,1-Dichloroethene	≤ 30.0	≤ 20.0
Chloroform	≤ 30.0	≤ 20.0
1,2-Dichloropropane	≤ 30.0	≤ 20.0
Toluene	≤ 30.0	≤ 20.0
Ethylbenzene	≤ 30.0	≤ 20.0

Table 13. Characteristic Ions

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d_4 (Surrogate)	65	102	--
Dichlorodifluoromethane	85	87	50, 101,103
Dibromofluoromethane	111	113	--
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	--
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	--
Acetone	43	58	--
Methylene chloride	84	49	51, 86
Tert-Butyl alcohol	59	74	--
Trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl-*tert*-butyl ether	73	41	57
Hexane	57	43	--
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	--
Tetrahydrofuran	42	71	--
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	--
Vinyl acetate	43	86	--
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Benzene	78	52	77
Trichloroethene	95	130***	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	--
Xylenes	106	91	--
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
Trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	41	76	78
Acetonitrile	41	40	--
Dichlorofluoromethane	67	69	--
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	--
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	--
2,2-Dichloropropane	77	97	--
Bromochloromethane	128	49	130
1,1-Dichloropropene	75	39	110
1,3-Dichloropropane	76	41	78
1-Chlorohexane	91	55	41
1,1,1,2-Tetrachloroethane	131	133	--
Bromobenzene	156	158	77
n-Propylbenzene	120	91	65
2-Chlorotoluene	126	91	65
1,3,5-Trimethylbenzene	105	120	77
4-Chlorobenzene	126	91	89
t-Butylbenzene	119	134	91
sec-Butylbenzene	134	105	--
4-Isopropyltoluene	119	134	91
n-Butylbenzene	91	92	134
1,2,4-Trichlorobenzene	180	182	--
Hexachlorobutadiene	225	227	223
Naphthalene	128	127	--
1,2,3-Trichlorobenzene	180	182	--
1,1,1-Trifluoro-2,2-Dichloroethane	83	133	--
1,2,3-Trimethylbenzene	105	120	91
1,2,4-Trimethylbenzene	105	120	119
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	85	87	--
1,2-Dichloro-1,1,2-Trifluoroethane	117	67	85
1,3,5-Trichlorobenzene	180	182	184
2,2,3-Trimethylbutane	57	43	85
2,2-Dimethylpentane	57	43	85
2,3-Dimethylpentane	56	71	73

Table 13. Characteristic Ions (cont.)

Compound	Primary*	Secondary	Tertiary
2,4-Dimethylpentane	43	57	85
2-Chloro-1,1,1-Trifluoroetane	118	83	69
2-Methylhexane	43	85	57
3,3-Dimethylpentane	43	71	--
3-Ethylpentane	43	70	71
3-Methylhexane	43	57	71
4-Chlorotoluene	126	91	89
2-Pentanone	43	86	--
Chlorotrifluoroethene	116	66	97
Cis-1,4-Dichloro-2-butene	53	75	89
Cyclohexane	56	84	55
Dimethyl Disulfide	94	79	45
Ethylene Dibromide	107	109	--
Ethylene Oxide	43	44	--
Isopropyl Alcohol	45	43	--
Methyl Acetate	43	74	59
Methylcyclohexane	55	83	41
m-Xylene & p-Xylene	91	106	77
n-Heptane	43	100	71
n-Nonyl Aldehyde	46	44	207
O-Xylene	106	91	--
Propene Oxide	58	43	57
Sec-Butyl Alcohol	45	59	--
Tert-amyl methyl ether	73	55	87
Tert-butyl ethyl ether	59	87	57
Tetrahydrothiophene	60	88	45

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-butanone, but m/z 72 must be present for positive identification.

*** Used as quantitation ion for method 624/624.1.

Table 14. State of Arizona ICV/CCV Quality Control Limits

QC Limits not specified in method	Default QC (method specified or laboratory historical if not specified)
CCV Non-CCC compounds	CCC limits (≤30%)
ICV	Same as CCV (≤30%)
Reporting Limit	Must be supported by low level initial calibration standard
LCS/LCSD	Lab historical
MS/MSD	Lab historical

NOTES:

1) Based on ADHS Rule A.A.C.R9-14-615.C.8. Director approved on June 29, 2005 for the labs to use default limits as an alternative to developing statistically derived limits.

Table 15. List 1 Poorly Performing Compounds

The laboratory's GC/MS group identified this list of compounds based on current and historical performance. The recovery performance was reviewed against full spike recovery data and method performance data, where available, to validate each compound as a "poor performer."

Acetone	1,2-Dichloro-1,1,2,2-tetrafluoroethane
Acetonitrile	Ethanol
Acrolein	Ethyl acetate
Acrylonitrile	Ethylene oxide
n-Butanol	2-Hexanone
2-Butanone (MEK)	Isobutyl alcohol
tert-Butyl alcohol	Isopropanol
Carbon disulfide	Methacrylonitrile
2-Chloroethyl vinyl ether	Methyl acetate
2-Chloro-1,1,1-trifluoroethane	4-Methyl-2-pentanone
Chlorotrifluoroethene	2-Nitropropane
cis-1,4-Dichloro-2-butene	2-Pentanone
trans-1,4-Dichloro-2-butene	2-Propanol
Dichlorodifluoromethane	Propionitrile
Dichlorofluoromethane	Tetrahydrofuran
1,2-Dibromo-3-chloropropane (DBCP)	Tetrahydrothiophene
1,2-Dichlorotetrafluoroethane	1,1,2-Trichloro-1,2,2-trifluoroethane
1,2-Dichloro-1,1,2-trifluoroethane (Freon 123a)	Trichlorofluoromethane
2,2-Dichloro-1,1,1-trifluoroethane	Vinyl acetate
1,4-Dioxane	

Table A-1. Method 624/624.1 Analytes and Reporting Limits, 5-mL Purge

Analytes	µg/L
Acrolein[1]	100
Acrylonitrile[1]	100
Benzene	5
Bromodichloromethane	5
Bromoform	5
Bromomethane	10
Carbon tetrachloride	5
Chlorobenzene	5
Chloroethane	10
2-Chloroethyl vinyl ether	5
Chloroform	5
Chloromethane	10
Dibromochloromethane	5
1,2-Dichlorobenzene	5
1,3-Dichlorobenzene	5
1,4-Dichlorobenzene	5
1,1-Dichloroethane	5
1,2-Dichloroethane	5
1,1-Dichloroethene	5
trans-1,2-Dichloroethene	5
1,2-Dichloropropane	5
cis-1,3-Dichloropropene	10
trans-1,3-Dichloropropene	5
Ethylbenzene	5
Methylene chloride	5
1,1,2,2-Tetrachloroethane	5
Tetrachloroethene	5
Toluene	5
1,1,1-Trichloroethane	5
1,1,2-Trichloroethane	5
Trichloroethene	5
Trichlorofluoromethane	15
Vinyl chloride	10

[1] Acrolein and Acrylonitrile have been added to the 624/624.1 analyte list in the EPA Method Update Rule, May 18, 2012. Analysis of these analytes by Method 624/624.1 as being regulatory compliant is dependent upon individual state approval of the MUR. Verify state status before analysis.

Table A-2. Method 624/624.1 QC Acceptance Criteria

Table 7 – LCS (Q), DOC (s and x̄), and MS/MSD (P and RPD) Acceptance Criteria [1]					
Analyte	**Range for Q (%)**	**Limit for s (%)**	**Range for x̄(%)**	**Range for P_1, P_2**	**Limit for RPD**
Acrolein	60-140	30	50-150	40-160	60
Acrylonitrile	60-140	30	50-150	40-160	60
Benzene	65-135	33	75-125	37-151	61
Bromodichloromethane	65-135	34	50-140	35-155	56
Bromoform	70-130	25	57-156	45-169	42
Bromomethane	15-185	90	D-206	D-242	61
Carbon tetrachloride	70-130	26	65-125	70-140	41
Chlorobenzene	65-135	29	82-137	37-160	53
Chloroethane	40-160	47	42-202	14-230	78
2-Chloroethylvinyl ether	D-225	130	D-252	D-305	71
Chloroform	70-135	32	68-121	51-138	54
Chloromethane	D-205	472	D-230	D-273	60
Dibromochloromethane	70-135	30	69-133	53-149	50
1,2-Dichlorobenzene	65-135	31	59-174	18-190	57
1,3-Dichlorobenzene	70-130	24	75-144	59-156	43
1,4-Dichlorobenzene	65-135	31	59-174	18-190	57
1,1-Dichloroethane	70-130	24	71-143	59-155	40
1,2-Dichloroethane	70-130	29	72-137	49-155	49
1,1-Dichloroethene	50-150	40	19-212	D-234	32
trans-1,2-Dichloroethene	70-130	27	68-143	54-156	45
1,2-Dichloropropane	35-165	69	19-181	D-210	55
cis-1,3-Dichloropropene	25-175	79	5-195	D-227	58
trans-1,3-Dichloropropene	50-150	52	38-162	17-183	86
Ethyl benzene	60-140	34	75-134	37-162	63
Methylene chloride	60-140	192	D-205	D-221	28
1,1,2,2-Tetrachloroethane	60-140	36	68-136	46-157	61
Tetrachloroethene	70-130	23	65-133	64-148	39
Toluene	70-130	22	75-134	47-150	41
1,1,1-Trichloroethane	70-130	21	69-151	52-162	36
1,1,2-Trichloroethane	70-130	27	75-136	52-150	45
Trichloroethene	65-135	29	75-138	70-157	48
Trichlorofluoromethane	50-150	50	45-158	17-181	84
Vinyl chloride	5-195	100	D-218	D-251	66

[1] Criteria were calculated using an LCS concentration of 20 µg/L

Q = Percent recovery in calibration verification/LCS (Section 8.4)
s = Standard deviation of percent recovery for four recovery measurements (Section 8.2.4)
x̄ = Average percent recovery for four recovery measurements (Section 8.2.4)
P = Percent recovery for the MS or MSD (Section 8.3.3)
D = Detected; result must be greater than zero

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D

Analyte	Associated IS	Associated Surrogate
1,1,1,2-Tetrachloroethane	2	2
1,1,1-TCA	1	1
1,1,2,2-Tetrachloroethane	3	3
1,1,2-TCA	1	4
1,1-DCA	1	1
1,1-DCE	1	1
1,1-Dichloropropene	1	4
1,2,3-Trichlorobenzene	3	3
1,2,3-Trichloropropane	3	3
1,2,4-Trichlorobenzene	3	3
1,2,4-Trimethylbenzene	3	3
1,2-DCA	1	4
1,2-DCB	3	3
1,2-Dibromo-3-chloropropane	3	3
1,2-Dichloropropane	1	4
1,2-EDB	2	2
1,3,5-Trimethylbenzene	3	3
1,3-DCB	3	3
1,3-Dichloropropane	2	2
1,4-DCB	3	3
1-Chlorohexane	2	2
2,2-Dichloropropane	1	1
2-Chlorotoluene	3	3
4-Chlorotoluene	3	3
Acetone	1	1
Benzene	1	4
Bromobenzene	3	3
Bromochloromethane	1	1
Bromodichloromethane	1	4
Bromoform	2	2
Bromomethane	1	1
Carbon Tetrachloride	1	4
Chlorobenzene	2	2
Chloroethane	1	1
Chloroform	1	1
Chloromethane	1	1
Cis-1,2-DCE	1	1
Cis-1,3-Dichloropropene	1	4
Dibromochloromethane	2	2
Dibromomethane	1	4
Dichlorodifluoromethane	1	1
Ethylbenzene	2	2
Hexachlorobutadiene	3	3
Isopropylbenzene	3	3
M,P-Xylene	2	2
Methylene Chloride	1	1
Methyl tert-butyl ether (MTBE)	1	1

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D (cont.)

Analyte	Associated IS	Associated Surrogate
MEK (2-Butanone)	1	1
MIBK (4-Methyl-2-Pentanone)	1	4
n-Butylbenzene	3	3
n-Propylbenzene	3	3
Napthalene	3	3
o-Xylene	2	2
p-Isopropyltoluene	3	3
sec-Butylbenzene	3	3
Styrene	2	2
TCE	1	4
tert-butylbenzene	3	3
Tetrachloroethene	2	2
Toluene	1	4
trans-1,2-DCE	1	1
trans-1,3-Dichloropropene	1	4
Trichlorofluoromethane	1	1
Vinyl Chloride	1	1
Chlorotrifluoroethene	1	1
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	1	1
2-Chloro-1,1,1-Trifluoroethane	1	1
Ethylene Oxide	1	1
Ethanol	4	1
Ethyl Ether	1	1
1,2-Dichloro-1,1,2-Trifluoroethane	1	1
Propene Oxide	1	1
1,1,1-Trifluoro-2,2-dichloroethane	1	1
Acrolein	1	1
Iodomethane	1	1
Carbon Disulfide	1	1
3-Chloro-1-propene	1	1
Acetonitrile	1	1
Methyl Acetate	1	1
2-Methyl-2-propanol (TBA)	4	1
Acrylonitrile	1	1
Hexane	1	1
Vinyl Acetate	1	1
Isopropyl Alcohol	4	1
Isopropyl Ether	1	1
2-Chloro-1,3-butadiene	1	1
Tert-butyl ethyl ether (ETBE)	1	1
Propionitrile	1	1
Ethyl Acetate	1	1
sec-Butyl Alcohol	4	1
Methacrylonitrile	1	1
Tetrahydrofuran	1	1
Cyclohexane	1	1
Tert-amyl methyl ether (TAME)	1	1
Isobutyl Alcohol	4	1
n-Butanol	4	1

Table 17
Associated Surrogates and Internal Standards for 8260B/C/D (cont.)

2-Pentanone	1	4
Methycyclohexane	1	4
Methy Methacrylate	1	4
1,4-Dioxane	1	4
2-Nitropropane	1	4
2-Chloroethyl vinyl ether (2-Cleve)	1	4
Ethyl Methacrylate	1	4
2-Hexanone	2	2
Tetrahydrothiophene	2	2
1-Chlorohexane	2	2
cis-1,4-Dichloro-2-butene	3	3
Cyclohexanone	2	2
Trans-1,4-Dichloro-2-butene	3	3
Surrogates	Associated IS	Associated Surrogate
Dibromofluoromethane	1	1
Toluene-d8	2	2
4-Bromofluorobenzene	3	3
1,2-DCA-d4	1	4
Internal Standards	Associated IS	Associated Surrogate
Fluorobenzene	1	-
Chlorobenzene-d_5	2	-
1,4-Dichlorobenzene	3	-
TBA-d_9	4	-
1,4-Dioxane-d_8*	5	-

* 1,4-Dioxane-d_8 is in the Internal Standard spiked into every sample but is not associated to any analytes due to poor stability. 1,4-Dioxane-d_8 must be set to “rejected” or “not needed” status in TALS.

In the event that a compound is not covered by this table is requested the associated internal standard/surrogate will be used as required by the method.

Table 18: 8260C (D) minimum response factors

Compound	Minimum response factor
1,1,1,2-Tetrachloroethane	0.010
1,1,1-Trichloroethane	0.100 (0.050)
1,1,2,2-Tetrachloroethane	0.300 (0.200)
1,1,2-Trichloro-1,2,2-Trifluoroethane	0.100 (0.050)
1,1,2-Trichloroethane	0.100 (0.200)
1,1-Dichloroethane	0.200 (0.300)
1,1-Dichloroethene	0.100 (0.060)
1,1-Dichloropropene	0.010
1,2,3-Trichlorobenzene	0.010 (0.400)
1,2,3-Trichloropropane	0.010
1,2,4-Trichlorobenzene	0.200
1,2,4-Trimethylbenzene	0.010 (0.400)
1,2-Dibromo-3-chloropropane	0.050 (0.010)
1,2-Dichlorobenzene	0.400 (0.600)
1,2-Dichloroethane	0.100 (0.070)
1,2-Dichloropropane	0.100 (0.200)
1,3,5-Trimethylbenzene	0.010
1,3-Dichlorobenzene	0.600 (0.500)
1,3-Dichloropropane	0.010
1,4-Dichlorobenzene	0.500 (0.600)
1,4-Dioxane	0.001
1-Chlorohexane	0.010
2-Chloroethyl vinyl ether	0.010
2,2-Dichloropropane	0.010
2-Butanone (MEK)	0.100 (0.010)
2-Chlorotoluene	0.010
2-Hexanone	0.100 (0.010)
2-Pentanone	0.001
3-Chloro-1-propene (Allyl Chloride)	0.010
2-Methyl-2-propanol (tert-Butyl alcohol)	0.001
4-Chlorotoluene	0.010
4-Isopropyltoluene	0.010
4-Methyl-2-pentanone	0.100 (0.030)
Acetone	0.050 (0.010)
Acrolein	0.001
Acrylonitrile	0.010

Compound	Minimum response factor
Benzene	0.500 (0.200)
Bromobenzene	0.010
Bromoform	0.100 (0.100)
Bromomethane	0.100 (0.010)
Carbon Disulfide	0.100 (0.100)
Carbon tetrachloride	0.100 (0.100)
Chlorobenzene	0.500 (0.400)
Chlorobromomethane	0.010 (0.100)
Chlorodibromomethane	0.100 (0.300)
Chloroethane	0.100 (0.010)
Chloroform	0.200 (0.300)
Chloromethane	0.100 (0.010)
cis-1,2-Dichloroethene	0.100 (0.200)
cis-1,3-Dichloropropene	0.200 (0.300)
Cyclohexane	0.100 (0.010)
Cyclohexanone	0.001
Dibromomethane	0.010
Dichlorobromomethane	0.200 (0.200)
Dichlorofluoromethane	0.010 (0.010)
Dichlorodifluoromethane	0.100
Ethylbenzene	0.100 (0.400)
Ethyl Ether	0.010
Ethyl methacrylate	0.010
Ethylene dibromide (EDB)	0.100 (0.200)
Hexachlorobutadiene	0.010
Hexane	0.010
Iodomethane	0.010
Isobutyl alcohol	0.001
Isopropylbenzene	0.100 (0.400)
m and p Xylenes	0.100 (0.200)
Methyl Acetate	0.100 (0.010)
Methylcyclohexane	0.100 (0.050)
Methylene chloride	0.100 (0.010)
Methyl tert-butyl ether (MTBE)	0.100 (0.100)
Naphthalene	0.010
n-Butylbenzene	0.010
n-Propylbenzene	0.010
o-Xylene	0.300 (0.200)

Compound	Minimum response factor
sec-Butylbenzene	0.010
Styrene	0.300 (0.200)
tert-Butylbenzene	0.010
Tetrachloroethene	0.200 (0.100)
Tetrahydrofuran	0.001
Toluene	0.400 (0.300)
trans-1,2-Dichloroethene	0.100 (0.100)
trans-1,3-Dichloropropene	0.100 (0.300)
trans-1,4-Dichloro-2-butene	0.010
Trichloroethene	0.200 (0.200)
Trichlorofluoromethane	0.100 (0.010)
Vinyl Acetate	0.010
Vinyl chloride	0.100 (0.010)
1,2-Dichloroethane-d4 (surr)	0.010
Toluene-d8 (surr)	0.010
4-Bromofluorobenzene (surr)	0.010
Dibromofluoromethane (surr)	0.010

8260B versus 8260C/D

Method 8260C/D does not have CCC or SPCC. In the ICV and CCV all target compounds must meet either the listed minimum response factor or a lab derived response factor and 20% drift. In the initial calibration for 8260C/D compliant work each compound must meet 20% RSD to be a valid calibration for that compound. 8260B and 8260C/D have different control limits for spikes and surrogates. See TALs for limits.

APPENDIX A

Modifications for Analysis of Soils Collected for the State of Alaska

1. Collection and Preservation Requirements

Preservation and Holding Time for Volatiles in Soil
Method 5035A for Alaska

Container/Contents[1]	Preservation	Holding time	Analysis
Vial containing methanol and TFT surrogate	Sample is extruded into pre-tared 4 oz jar, containing 25 mL of methanol spiked with 2.5 ppm (ug/mL) of α, α, α-trifluorotoluene, cooled to ≤ 6°C and frozen upon receipt at laboratory.	14 days	Medium Level

Sample weights are calculated in the laboratory by adding the received weight of the sample into the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

2. Sample Preparation for Medium-Level Analysis – Field Preserved, AK method

a. Fill a 40 mL VOA vial with reagent water ~ 42 mL (no head space), and remove 1000 µL of water using a volumetric pipette or syringe.

b. Add 1050 µL of methanol extract to the vial and immediately cap. Invert the vial to ensure that there is no air bubble larger than 4 mm present. If a > 4 mm air bubble is present, re-prepare the sample.

c. Load the sample in the auto sampler and proceed to analyze against the methanol calibration curve.

d. As with water samples, surrogate and internal standard solutions are added by the autosampler (see Tables 7 and 7A in the main body of this SOP). The surrogate α, α, α-trifluorotoluene is added to the samples at the time of sampling. Recoveries for this surrogate will be reported in addition to recoveries for the surrogate compounds added at the time of analysis.

e. Prepare laboratory control samples by filling a 40 mL VOA vial with reagent water, and remove 1000 µL of water using a volumetric pipette or syringe. Add reagents as needed plus sufficient methanol for a total methanol volume of 1050 µL. The recommended concentration for the LCS is the same as the Level 5 of the initial calibration curve.

f. Remove a portion of the methanol extract for each sample and store in a clean Teflon-capped vial with no headspace at ≤ 6 °C until analysis. Duplicate aliquots of the methanol extract should be taken and stored.

3. Percent Moisture Correction for Soils from the State of Alaska

A percent moisture correction is required for soil samples submitted from the state of AK to adjust the extraction final volume in order to allow for the miscible solvent effects. The following

formula is used to determine the corrected final volume. This calculation is performed in the AK Methanol Volume Correction spreadsheet stored on G:\QA\Edit\FORMS\GCMS.

$$V_t = [V_m + (M * W_s/100)]$$

Where:

V_t = final extract volume, corrected for moisture (mL)

V_m = volume methanol used for extraction (mL)

M = moisture content of the sample (%)

W_s = aliquot of sample extracted (g)

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Dichlorodifluoromethane	75-71-8	80
Chloromethane	74-87-3	40
Bromomethane	74-83-9	40
Vinyl chloride	75-01-4	40
Chloroethane	75-00-3	40
n-Butanol	71-36-3	800
Trichlorofluoromethane	75-69-4	40
Acrolein	107-02-8	200
Acetone	67-64-1	400
Trichlorotrifluoroethane	76-13-1	400
Iodomethane	74-88-4	500
Carbon disulfide	75-15-0	40
Methylene chloride	75-09-2	40
tert-Butyl alcohol	75-65-0	800
1,1-Dichloroethene	75-35-4	40
1,1-Dichloroethane	75-34-3	40
trans-1,2-Dichloroethene	156-60-5	40
Acrylonitrile	107-13-1	400
Methyl *tert*-butyl ether (MTBE)	1634-04-4	200
Hexane	110-54-3	400
cis-1,2-Dichloroethene	156-59-2	40
1,2-Dichloroethene (Total)	540-59-0	40
Tetrahydrofuran	109-99-9	80
Chloroform	67-66-3	40
1,2-Dichloroethane	107-06-2	40
Dibromomethane	74-95-3	40
2-Butanone	78-93-3	160
1,4-Dioxane	123-91-1	2,000
1,1,1-Trichloroethane	71-55-6	40
Carbon tetrachloride	56-23-5	40
Bromodichloromethane	75-27-4	40
1,2-Dichloropropane	78-87-5	40
Isopropyl Alcohol	67-63-0	1,000
Isopropyl ether	108-20-3	200

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
cis-1,3-Dichloropropene	10061-01-5	40
Trichloroethene	79-01-6	40
Dibromochloromethane	124-48-1	40
1,2-Dibromoethane	106-93-4	40
1,2,3-Trichloropropane	96-18-4	40
1,1,2-Trichloroethane	79-00-5	40
Benzene	71-43-2	16
Ethylmethacrylate	97-63-2	80
trans-1,3-Dichloropropene	10061-02-6	40
Bromoform	75-25-2	40
4-Methyl-2-pentanone	108-10-1	160
2-Hexanone	591-78-6	160
Tetrachloroethene	127-18-4	40
Toluene	108-88-3	40
1,1,2,2-Tetrachloroethane	79-34-5	40
2-Chloroethyl vinyl ether	110-75-8	80
Vinyl acetate	108-05-4	80
Chlorobenzene	108-90-7	40
Ethylbenzene	100-41-4	40
Styrene	100-42-5	40
trans-1,4-Dichloro-2-butene	110-57-6	400
m- and p-Xylenes	179601-23-1	80
o-Xylene	95-47-6	40
Total xylenes	1330-20-7	80
1,3-Dichlorobenzene	541-73-1	40
1,4-Dichlorobenzene	106-46-7	40
1,2-Dichlorobenzene	95-50-1	40
2,2-Dichloropropane	590-20-7	40
Bromochloromethane	74-97-5	40
1,1-Dichloropropene	563-58-6	40
1,3-Dichloropropane	142-28-9	40
1-Chlorohexane	544-10-5	80
1,1,1,2-Tetrachloroethane	630-20-6	40

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
Isopropylbenzene	98-82-8	40
Bromobenzene	108-86-1	40
n-Propylbenzene	103-65-1	40
2-Chlorotoluene	95-49-8	40
4-Chlorotoluene	106-43-4	40
1,3,5-Trimethylbenzene	108-67-8	40
tert-Butylbenzene	98-06-6	40
1,2,4-Trimethylbenzene	95-63-6	40
sec-Butylbenzene	135-98-8	40
4-Isopropyltoluene	99-87-6	40
n-Butylbenzene	104-51-8	40
1,2-Dibromo-3-chloropropane	96-12-8	200
1,2,4-Trichlorobenzene	120-82-1	40
Naphthalene	91-20-3	40
Hexachlorobutadiene	87-68-3	40
1,2,3-Trichlorobenzene	87-61-6	40
Propionitrile	107-12-0	400
Cylcohexanone	108-94-1	1,600
Methyl methacrylate	80-62-6	80
Acetonitrile	75-05-8	400
Methacrylonitrile	126-98-7	400
1,2-Dichloro-1,1,2,2-Tetrafluoroethane	76-14-2	160
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	160
2-Pentanone	107-87-9	600
cis-1,4-Dichloro-2-butene	1476-11-5	400
Cyclohexane	110-82-7	40
Methyl acetate	79-20-9	200
Methylcyclohexane	108-87-2	160
2-Chloro-1,3-butadiene	126-99-8	80
2-Methyl-2-propanol	75-65-0	800

Table Ap-1. TestAmerica 8260 Reporting Limits – AK Soils

Compound	CAS Number	Medium Soil µg/Kg
tert-Butyl ethyl ether	637-92-3	80
1,2,3-Trimethylbenzene	526-73-8	40
Ethyl acetate	141-78-6	80
Ethyl ether	60-29-7	200
Isobutyl alcohol	78-83-1	800
Dichlorofluoromethane	75-43-4	120
Tetrahydrothiophene	110-01-0	40

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

Table Ap-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
1,1,1,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,1-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2,2-Tetrachloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,1-Dichloroethene	20	40	80	200	600	2000	4000	8000
1,1-Dichloropropene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,3-Trichloropropane	20	40	80	200	600	2000	4000	8000
1,2,4-Trichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2,4-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dibromo-3-chloropropane	20	40	80	200	600	2000	4000	8000
1,2-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,3,5-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,3-Dichloropropane	20	40	80	200	600	2000	4000	8000
1,4-Dichlorobenzene	20	40	80	200	600	2000	4000	8000
1,4-Dioxane	1000	2000	4000	10000	30000	100000	200000	400000
1-Chlorohexane	20	40	80	200	600	2000	4000	8000
2,2-Dichloropropane	20	40	80	200	600	2000	4000	8000
2-Butanone (MEK)	80	160	320	800	2400	8000	16000	32000
2-Chloro-1,3-butadiene (chloroprene)	20	40	80	200	600	2000	4000	8000
2-Chlorotoluene	20	40	80	200	600	2000	4000	8000
2-Hexanone	80	160	320	800	2400	8000	16000	32000
2-Methyl-2-propanol (tert-Butyl alcohol)	400	800	1600	4000	12000	40000	80000	160000
4-Chlorotoluene	20	40	80	200	600	2000	4000	8000
4-Isopropyltoluene	20	40	80	200	600	2000	4000	8000
4-Methyl-2-pentanone	80	160	320	800	2400	8000	16000	32000
Acetone	80	160	320	800	2400	8000	16000	32000
Acetonitrile	200	400	800	2000	6000	20000	40000	80000
Acrolein	200	400	800	2000	6000	20000	40000	80000
Acrylonitrile	200	400	800	2000	6000	20000	40000	80000

Table Ap-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
Benzene	20	40	80	200	600	2000	4000	8000
Bromobenzene	20	40	80	200	600	2000	4000	8000
Bromoform	20	40	80	200	600	2000	4000	8000
Bromomethane	20	40	80	200	600	2000	4000	8000
Carbon tetrachloride	20	40	80	200	600	2000	4000	8000
Chlorobenzene	20	40	80	200	600	2000	4000	8000
Chlorobromomethane	20	40	80	200	600	2000	4000	8000
Chlorodibromomethane	20	40	80	200	600	2000	4000	8000
Chloroethane	20	40	80	200	600	2000	4000	8000
Chloroform	20	40	80	200	600	2000	4000	8000
Chloromethane	20	40	80	200	600	2000	4000	8000
cis-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
cis-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Cyclohexanone	20	40	80	200	300	1000	2000	4000
Dibromomethane	20	40	80	200	600	2000	4000	8000
Dichlorobromomethane	20	40	80	200	600	2000	4000	8000
Dichlorodifluoromethane	20	40	80	200	600	2000	4000	8000
Ethylbenzene	20	40	80	200	600	2000	4000	8000
Ethylene dibromide (EDB)	20	40	80	200	600	2000	4000	8000
Hexachlorobutadiene	20	40	80	200	600	2000	4000	8000
Iodomethane	20	40	80	200	600	2000	4000	8000
Isopropyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Isopropyl ether	100	200	400	1000	3000	10000	20000	40000
Isopropylbenzene	20	40	80	200	600	2000	4000	8000
m- and p-Xylenes	40	80	160	400	1200	4000	8000	16000
Methacrylonitrile	200	400	800	2000	6000	20000	40000	80000
Methylene chloride	20	40	80	200	600	2000	4000	8000
Naphthalene	20	40	80	200	600	2000	4000	8000
n-Butanol	600	1200	2400	6000	18000	60000	120000	240000
n-Butylbenzene	20	40	80	200	600	2000	4000	8000
n-Propylbenzene	20	40	80	200	600	2000	4000	8000
o-Xylene	20	40	80	200	600	2000	4000	8000
Propionitrile	200	400	800	2000	6000	20000	40000	80000
sec-Butylbenzene	20	40	80	200	600	2000	4000	8000
Styrene	20	40	80	200	600	2000	4000	8000
tert-Butylbenzene	20	40	80	200	600	2000	4000	8000

Table Ap-2								
Calibration Levels for 8260, 5035FM_AK (ug/Kg)								
Compound	**Level 1**	**Level 2**	**Level 3**	**Level 4**	**Level 5**	**Level 6**	**Level 7**	**Level 8**
Tetrachloroethene	20	40	80	200	600	2000	4000	8000
Tetrahydrothiophene	20	40	80	200	600	2000	4000	8000
Toluene	20	40	80	200	600	2000	4000	8000
trans-1,2-Dichloroethene	20	40	80	200	600	2000	4000	8000
trans-1,3-Dichloropropene	20	40	80	200	600	2000	4000	8000
Trichloroethene	20	40	80	200	600	2000	4000	8000
Trichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Vinyl chloride	20	40	80	200	600	2000	4000	8000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Table Ap-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
1,1,1-Trifluoro-2,2-dichloroethane	20	40	80	200	600	2000	4000	8000
1,1,2-Trichloro-1,2,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
1,2,3-Trimethylbenzene	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2,2-tetrafluoroethane	20	40	80	200	600	2000	4000	8000
1,2-Dichloro-1,1,2-trifluoroethane	20	40	80	200	600	2000	4000	8000
2-Chloroethy vinyl ether	20	40	80	200	600	2000	4000	8000
2-Nitropropane	20	40	80	200	600	2000	4000	8000
2-Pentanone	80	160	320	800	2400	8000	16000	32000
3-Chloro-1-propene (Allyl chloride)	20	40	80	200	600	2000	4000	8000
Carbon disulfide	20	40	80	200	600	2000	4000	8000
cis-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Cyclohexane	20	40	80	200	600	2000	4000	8000
Dichlorofluoromethane	20	40	80	200	600	2000	4000	8000
Ethyl acetate	40	80	160	400	1200	4000	8000	16000
Ethyl ether	20	40	80	200	600	2000	4000	8000
Ethyl methacrylate	40	80	160	400	1200	4000	8000	16000
Ethylene oxide	2500	5000	10000	25000	75000	250000	500000	1000000

Table Ap-3: 5035FM_AK Calibration Levels (µg/Kg)[1]
(Standards: MV-Supp Std and MV-2 Cleve)

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Hexane	20	40	80	200	600	2000	4000	8000
Isobutyl alcohol	400	800	1600	4000	12000	40000	80000	160000
Methyl acetate	100	200	400	1000	3000	10000	20000	40000
Methylcylcohexane	20	40	80	200	600	2000	4000	8000
Methyl methacrylate	40	80	160	400	1200	4000	8000	16000
Methyl *tert*-butyl ether (MTBE)	20	40	80	200	600	2000	4000	8000
Propene oxide	400	800	1600	4000	12000	40000	80000	160000
sec-Butyl alcohol	600	1200	2400	6000	18000	60000	120000	240000
tert-Amyl methyl ether	100	200	400	1000	3000	10000	20000	40000
tert-Butyl ethyl ether	100	200	400	1000	3000	10000	20000	40000
Tetrahydrofuran	40	80	160	400	1200	4000	8000	16000
trans-1,4-dichloro-2-butene	20	40	80	200	600	2000	4000	8000
Vinyl acetate	40	80	160	400	1200	4000	8000	16000

[1]Standards are spiked at all levels. A minimum of 5 points are used for each calibration model. Low points below the RL are routinely dropped and the high point might also be dropped for some analytes.

Attachment 1

Gas Standards Tracking Log

Gas Standards Tracking Log

SOP ID: DV-MS-0010

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Standard ID#	Label ID#	Open Date	Analyst	Discard Date	Analyst

G:\MsVoaSem\Hidden logs\gas standard tracking.pdf 1/2011 Rev. 0

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GC/MS Analysis Based On Methods 8270C and 625/625.1

Approvals:

3/3/20	3/4/2020
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1.0 Scope and Application

1.1 This method is based upon EPA Method SW846 8270C, and is applicable to the determination of the concentration of semivolatile organic compounds in extracts prepared from solid and aqueous matrices.

1.1.1 The modifications presented in Appendix A may be followed for analysis of wastewater by Method 625/625.1.

1.1.2 The modifications presented in Appendix B may be followed for analysis of wastewater using method 8270 (Best Practices). The best practices method provides for lower reporting limits in aqueous samples.

1.1.3 Direct injection of a sample may be used in limited applications.

1.1.4 Refer to Tables 1, 2 and 3 for the list of compounds applicable for this method. Note that the compounds are listed in approximate retention time order. This method may be amenable to additional compounds. If non-routine analytes are required, they must be validated by the procedures described in Section 13 before sample analysis.

1.2 The following compounds may require special treatment when being determined by this method:

1.2.1 Benzidine can be subject to oxidative losses during solvent concentration and exhibits poor chromatography.

1.2.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

1.2.3 N-Nitrosodimethylamine may be difficult to separate from the solvent under the instrument conditions in the method.

1.2.4 N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.

1.2.5 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

1.2.6 Pyridine may perform poorly at the injection port temperatures described in the method.

1.2.7 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3/4-methylphenol.

1.2.8 Hexachlorophene is not quantitatively reliable by this method. Hexachlorophene is only analyzed using a single point standard which provides semi-quantitative results.

1.2.9 Kepone should be analyzed by GC/ECD.

1.2.10 The calibration standard includes azobenzene. Azobenzene is a decomposition product of 1, 2-Diphenylhydrazine. In the calibrations standard both compounds are included, however the concentration for 1, 2-Diphenylhydrazine is adjusted to compensate for the decomposition using a factor of 1.011 determined as follows:

A/B where A = the molecular weight of Azobenzene and B = the molecular weight of 1, 2-diphenylhydrazine.

1.3 The standard reporting limit (SRL) of this method for determining an individual compound is approximately 0.33 – 3.3 mg/kg (wet weight) for soil/sediment samples, 1 - 200 mg/kg for wastes (dependent on matrix and method of preparation), and 4 - 200 µg/L for groundwater samples. Some compounds have higher reporting limits. Refer to Tables 1, 2, and 3 for specific SRLs. Reporting limits will be proportionately higher for sample extracts that require dilution.

2.0 Summary of Method

2.1 Aqueous samples are extracted with methylene chloride using a continuous extractor or a separatory funnel. TestAmerica Denver performs extractions using a one liter sample aliquot or a 250 mL sample aliquot. The extract is dried, concentrated to 1 mL and analyzed by GC/MS.

2.1.1 Samples extracted using 250 mL are analyzed following the Large Volume Injection (LVI) procedures described in this SOP.

2.1.2 LVI is used only for aqueous samples. It is utilized to maintain reporting limits while reducing the initial volume of the sample required for extraction. The extraction procedure is the same with the only adjustments to sample size, spike concentrations and volume of extraction solvent.

2.2 Solid samples are extracted with methylene chloride / acetone using sonication or Soxhlet extraction. The extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS.

2.3 Waste dilution is used for samples that are miscible with the solvent.

2.4 Extraction and cleanup procedures are detailed in the following SOPs:

DV-OP-0006	Extraction of Aqueous Samples by Separatory Funnel, SW846 3510C and EPA 600 Series
DV-OP-0007	Concentration and Cleanup of Organic Extracts, SW846 3510C, 3520C, 3540C, 3546, 3550B, 3550C, 3620C, 3660B, 3665A and EPA 600 Series
DV-OP-0008	Extraction of Aqueous Samples by Continuous Liquid/Liquid Extraction (CLLE) by Method SW-846 3520C and Method 625/625.1
DV-OP-0012	Waste Dilution Preparation for Non-Aqueous Waste Samples, SW-846 3580A

DV-OP-0016 Ultrasonic Extraction of Solid Samples, SW846 3550B and 3550C

2.5 Sample extracts are analyzed by capillary GC/MS. The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) where the analytes are separated and then introduced into the mass spectrometer. The mass spectra are produced by electron impact. Qualitative identification is accomplished by comparing the extract spectra with the spectra of standards. Identification of target analytes of the analytes in the extract is performed using the retention time and the relative abundance of characteristic ions. Quantitative analysis is performed using the internal standard technique, with a single characteristic ion and a minimum of a five-point calibration curve.

2.6 For LVI the GC/MS analysis is the same with the exception of the injection volume and standard concentrations.

3.0 Definitions

3.1 CCC (Calibration Check Compounds) - A subset of target compounds used to evaluate the calibration stability of the GC/MS system. A maximum percent deviation of the CCCs is specified for calibration acceptance.

3.2 SPCC (System Performance Check Compounds) - Target compounds designated to monitor chromatographic performance, sensitivity, and compound instability or degradation on active sites. Minimum response factors are specified for acceptable performance.

3.3 Batch - The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The Quality Control batch must contain a matrix spike / matrix spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank (MB). If it is not possible to prepare both an MS and MSD due to limitations of sample amount, then a duplicate LCS should be prepared and analyzed. The RPD between the LCS and LCSD must be less than or equal to the RPD limit established for the MS/MSD.

3.3.1 Batches are defined at the sample preparation stage. Batches should be kept together through the whole analytical process to the extent possible, but it is not mandatory to analyze prepared extracts on the same instrument or in the same sequence. Refer to the TestAmerica QC Program document (DV-QA-003P) for further details of the batch definition.

3.3.2 Some clients or programs (e.g., DoD) specify the frequency of the MS/MSD relative to the field sampling and do not link it to the QC batch in the lab. In these cases an LCSD is not performed in the absence of batch MS/MSD if the batch only contains DoD samples.

3.4 Method Blank (MB) - An analytical control consisting of all reagents, internal standards and surrogate standards that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background and reagent contamination.

3.5 Laboratory Control Sample (LCS) - A blank matrix (reagent water or Ottawa Sand) spiked with the analytes of interest that is carried through the entire analytical procedure. Analysis

of this sample with acceptable recoveries of the spiked analytes demonstrates that the laboratory techniques for this method are acceptable.

3.6 Matrix Spike (MS) – An aliquot of a matrix (water or soil) fortified (spiked) with known amounts of specific analytes and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

3.7 Matrix Spike Duplicate (MSD) - A second aliquot of the same sample as the matrix spike (above) that is spiked in order to determine the precision of the method by measuring the relative percent difference (RPD) between the MS and MSD results.

3.8 Surrogates - Organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Surrogate recoveries are used to assess accuracy, method performance, and extraction efficiency for each sample analyzed. Each sample, blank, LCS, MS, and MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits for the project.

3.9 Lower Limit of Quantitation (LLOQ) – The lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be $\geq$ the lowest point in the calibration curve. The LLOQ is equivalent to the standard reporting limit. The required LLOQ verification is performed at a concentration of 1-2 times the LLOQ (or RL).

4.0 Interferences

4.1 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the sample. Cleanup procedures may help to eliminate select interferences, as follows:

4.1.1 Method 3660B, Sulfur Cleanup – If a sulfur peak is detected, copper or mercury can be used to treat the extract and remove the sulfur.

4.1.2 Other, more aggressive cleanup procedures listed in SW-846 may be used for select compounds listed in this procedure, but may cause degradation of some of the more reactive compounds. Consult with a technical expert in the laboratory for more difficult interference problems.

NOTE: Details concerning cleanup steps are described in the organic extraction SOPs (see Section 2.4).

4.2 Contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts may cause method interferences. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section (Section 9.2). Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples; then take corrective action to eliminate the problem.

4.3 The use of high purity reagents, solvents, and gases helps to minimize interference problems.

4.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed with solvent between samples. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of solvent to check for cross contamination.

4.5 Phthalate contamination is commonly observed in this analysis and its occurrence should be carefully evaluated as an indicator of a contamination problem in the sample preparation step of the analysis.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

NOTE: Latex and vinyl gloves provide no protection against the organic solvents used in this method. Nitrile or similar gloves must be used.

5.3.2 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.3.3 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.3.4 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power before performing any maintenance.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating.

NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Materials with Significant or Serious Hazard Rating

Material	Hazards	Exposure Limit (1)	Signs and Symptoms of Exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system: an analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to the source.

6.2 Column: 30 m x 0.25 mm I.D., 0.5-µm film thickness fused-silica capillary column coated with 5% diphenyl/95% dimethyl polysiloxane (Restek Rtx®-5MS or equivalent). Alternate columns are acceptable if they provide acceptable performance.

6.3 Mass Spectrometer: Capable of scanning from 35 to 500 u (previously "amu") every one second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) that meets all of the criteria in Table 5 when 25 ng of the GC/MS tuning standard is injected through the GC.

6.4 Autosampler: HP7683 Autosampler or equivalent, with appropriate sample trays.

6.5 GC/MS Interface: Any GC-to-MS interface that gives acceptable calibration points and achieves acceptable tuning performance criteria may be used.

6.6 Data System: A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIH Mass Spectral Library is recommended.

6.7 Analytical Syringe: 5 µL or 10 µL Hamilton Laboratory grade syringes or equivalent. The 5 µL syringe is used for the Agilent ALS to be able to inject 0.5 µL and the 10 µL syringe is commonly used for the Large Volume Injection.

6.8 Sample Aliquot Equipment: Laboratory grade syringes and pipettes as appropriate to dilute and aliquot extracts. Also, amber glass aliquot vials with polytetrafluoroethylene (PTFE)-lined screw or crimp caps and suitable for use with the autosampler.

6.9 Carrier gas: Ultra high-purity helium.

6.10 Injection port liners: Restek # 23303.5 or equivalent, split/splitless, single taper gooseneck with glass wool

6.11 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls (or current revision) for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Methylene Chloride, equivalent to pesticide quality or better, used to dilute samples as needed.

7.2 Stock standards are received from the vendor in multiple mixtures for each set of calibration standards, for both primary ICAL standards and second source verification standards. Other standards may be used to accommodate client-specific target lists or reporting limit requirements. Equivalent standards to those listed may be used if the standards specified below are not available.

7.2.1 Surrogate Standards:

Restek 567685 Surrogates at 5000 µg/mL

7.2.2 Internal Standards:

Restek 567684 Internal Standards at 2000 µg/mL

7.2.3 HSL (Primary) Standards:

Restek 567672 Mega Mix at 1000 µg/mL

Restek 567673 Amine Mix at 2000 µg/mL

Restek 567674 Benzoic Acid at 2000 µg/mL

Restek 567675 Indene at 2000 µg/mL

Restek 568023 Famphur at 2000 µg/mL

Restek 568033 HSL Custom Mix at 4000 µg/mL

Restek 568038 N-Nitrosodiphenylamine at 2000 µg/mL

7.2.4 AP9 Standards:

Restek 567678 Supplemental Standard 1 at 1000 µg/mL

Restek 567679 List 2 Standard 2 at 1000 µg/mL

Restek 567680 List 2 Standard 3 at 2000 µg/mL

Restek 567681 List 2 Standard 4 at 1000 µg/mL

Restek 567682 List 2 Standard 5 at 2000 µg/mL

Accustandard H-173N Dibenz[a,j]acridine, neat

7.2.5 Benzaldehyde Standards:

Restek 567677 Benzaldehyde at 2000 µg/mL

7.2.6 Hexachlorophene Standards:

Restek 31811 Hexachlorophene at 2000 µg/mL

7.2.7 Custom Standards:

Restek 568748 Custom Stock 1 at 2000 µg/mL

Restek 568749 Custom Stock 2 at 2000 µg/mL

7.2.8 Refinery Standards:

Supelco 21579195B Refinery Stock at 2000 µg/mL

SPEX S1200 Dibenzo(a,e)pyrene

7.2.9 PHE Standards:

Sigma Aldrich P23954 1,3-Phenylenediamine, neat

Supelco 442315 2,4-Xylidine, neat

Sigma Aldrich A88182 o-Anisidine, neat

Sigma Aldrich 103284 5-Methyl-o-Anisidine, neat

7.3 A minimum of seven calibration standards are typically prepared. Refer to Tables 10 and 11 for common calibration levels of the HSL and Appendix 9 analytes by the standard method. Other calibration compounds and levels may be used, depending on client requests and instrument capability, but the low standard must support the reporting limit and the high standard defines the range of the calibration. The low point should be at or below the reporting limit. A minimum five-point calibration curve is required when average response factors or linear regression curve fitting is used. Six calibration points are required for second-order curve fits. Table 12 provides calibration levels for many of the non-routine compounds listed in Table 3. Tables LVI-1, LVI-2 and LVI-3 provide calibration levels for LVI.

7.4 Initial Calibration Verification (ICV) Standards: For each curve, containing all components from a second source (an alternate vendor, a unique lot from the same vendor, or the same source but prepared by an alternate analyst). The ICV concentration may vary on a per-compound basis, but is usually prepared at 80 or 100 µg/mL concentration for full scan or 25 µg/mL for LVI. The Initial Calibration Verification must be ≤ ½ the concentration of the highest calibration standard for each analyte.

7.5 Continuing Calibration Verification (CCV) Standards: Also prepared for each curve and containing all the same components as was calibrated for, from the same vendor source as the calibration standards (Section 7.2). The CCV is usually prepared at the 80 µg/mL concentration for full scan analysis, and 20 µg/mL for LVI. It must be ≤ ½ the concentration of the highest calibration standard for each analyte.

7.6 An internal standard (IS) solution is prepared. Compounds in the IS Mix are acenaphthene-d_{10}, chrysene-d_{12}, 1,4-dichlorobenzene-d_4, naphthalene-d_8, perylene-d_{12}, and phenanthrene-d_{10}.

7.6.1 Internal standards are added to all standards and extracts to result in a final concentration of 40 µg/mL. For example, if the volume of an extract aliquot used was 200 µL, 20 µL of a 400 µg/mL internal standard solution would be added to the aliquot for the standard analysis. See Appendix B for the levels used for the 8270 best practice method.

7.7 Surrogate Standard Spiking Solution: Prepare as indicated in the extraction SOPs (refer to Section 2.4 for extraction SOP numbers). Concentration levels for surrogate compounds are listed in Table 9.

Acid Surrogates	**Base Surrogates**
2-Fluorophenol	2-Fluorobiphenyl
2,4,6-Tribromophenol	Terphenyl-d_4
Phenol-d_5	Nitrobenzene-d_5
2-Chlorophenol-d_4[1]	1,2-Dichlorobenzene-d_4[1]

[1] These two surrogates are in the mix but are not evaluated or reported.

7.8 GC/MS Tuning Standard: A methylene chloride solution containing 50 µg/mL of decafluorotriphenylphosphine (DFTPP) is prepared. Pentachlorophenol, benzidine, and DDT should also be included in the Tuning Standard at 50 µg/mL.

7.9 Laboratory Control Spiking Solution: Prepare as indicated in the SOP DV-OP-0020. The reported components of each LCS may vary and are selected by the client from the list of target analytes. For further information and to see current spike volumes and spiking solution concentrations see WI-DV-009 and the LIMS Reagent module.

7.10 Matrix Spike Solution: Prepare as indicated in the SOP DV-OP-0020. The matrix spike compounds and levels are the same as the LCS compounds.

7.11 The standards listed in sections 7.1 to 7.10 must be stored at -10°C to -20°C if it can be demonstrated that analytes do not fall out of solution at these temperatures. If not stable, the standards should be stored between 0°C and 6°C. The standard stock solutions expire after one year from preparation date or at the earliest expiration date assigned by the vendor to any parent standard, whichever is earlier. The continuing calibration standard should be replaced when there are visible signs of degradation or when the standard fails to meet QC criteria. The continuing calibration standard is stored at -10°C to -20°C. All standards must be warmed to room temperature before injecting for analysis.

8.0 Sample Collection, Preservation, Shipment and Storage

Matrix	Sample Container	Min. Sample Size	Preservation	Extraction Holding Time	Analysis Holding Time	Reference
Water	1 liter amber	1 liter	Cool, ≤6°C and not frozen	7 Days	40 Days from extraction	40 CFR Part 136.3, SW-846
Water[1]	250 mL amber	250 mL	Cool, ≤6°C and not frozen	7 Days	40 Days from extraction	40 CFR Part 136.3, SW-846
Soil	4 oz Jar	30 grams	Cool, <6°C and not frozen	14 Days	40 Days from extraction	SW-846

[1] Samples extracted using 250 mL are analyzed following the LVI procedure.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, Quality Assurance Program.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, Requirements for Federal Programs. DoD QSM 5.0 requirements for GCMS Methods are listed in Table 15 of this SOP.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

NOTE: QC Criteria specific to the DoD/DOE QSM are described in Table 15.

9.2 Method Blank (MB)

For aqueous sample batches, the method blank is reagent water; for solid sample batches, the method blank is clean sand. In either case, the method blank is free of the analytes of interest and is spiked with the surrogates. At least one method blank must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than ½ of the reporting limit or less than 10% of the analyte concentration found in the associated samples, whichever is higher. When a compound is above ½ the reporting limit a NCM needs to be completed.

NOTE: All programs require that the maximum blank concentration must be less than one-half of the reporting limit or less than 10% of the lowest sample concentration.

Corrective Action: Re-preparation and reanalysis of all samples associated with an unacceptable method blank. If the analyte was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

9.3 Instrument Blank

Instruments must be evaluated for contamination during each 12-hour analytical run. This may be accomplished by analysis of a method blank. If a method blank is not available, an instrument blank must be analyzed. An instrument blank consists of methylene

chloride with the internal standards added. It is evaluated in the same way as the method blank. (See Section 9.2.)

9.4 Laboratory Control Sample (LCS)

The LCS is prepared using reagent water for aqueous methods and Ottawa sand for solid sample methods. A laboratory control sample (LCS) is prepared and analyzed with every batch of samples. The LCS is spiked with the compounds as described in Section 7.9. Method 625/625.1 and the DoD/DOE QSM require that all target analytes be spiked. The compounds are typically spiked at a concentration equivalent to 80 µg/L, depending on the analyte. The spike amount must be at or below the midpoint of the calibration curve. (The LVI method uses the same concentration of analytes for the LCS.) Ongoing monitoring of the LCS provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Acceptance Criteria: All analytes must be within established control limits. See Quality Assurance Program DV-QA-003P for details on establishing control limits.

NOTE: Only use data associated with acceptable Calibration and QC samples for 625/625.1 compliance samples.

Corrective Action: If any analyte in the LCS is outside the laboratory-established historical control limits or project-specific control limits, as applicable, corrective action must occur. Corrective action may include re-extraction and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS. This type of justification should be documented and reviewed with the client before reporting.

- If re-extraction and reanalysis of the batch are not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in the narrative to provide further documentation.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

The matrix spike is a second aliquot of one of the samples in the batch. The matrix spike duplicate is a third aliquot of the same sample. The MS and MSD are spiked with the same analytes as the LCS (see Sections 7.9 and 7.10). An MS/MSD pair is prepared and analyzed with every batch of samples. Method 625/625.1 and the DoD/DOE QSM require that all target analytes be spiked.

Acceptance Criteria: The percent recovery (%R) must fall within either historical limits or project-specific limits, as applicable. The relative percent difference (RPD) between the MS and MSD results should be less than or equal to the established historical or project-specific limit. See Quality Assurance Program Policy DV-QA-003P for details on establishing control limits.

NOTE: DoD/DOE QSM 5 limits apply to projects performed under this program.

NOTE: Only use data associated with acceptable Calibration and QC samples for 625/625.1 compliance samples.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with

the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).

- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: Only use data associated with acceptable Calibration and QC samples for 625/625.1 compliance samples.

9.6 Surrogates

9.6.1 Each sample, blank, and QC sample is spiked with the surrogate standards. Surrogate compounds must be spiked at 100 µg/L. Compounds in the Primary Standard were each assigned to a surrogate based on the AFCEE QAPP, or if not specified therein, the retention time proximity to the nearest surrogate. The compounds routinely included in the surrogate spiking solution, along with their routine concentrations, are listed in Table 9. For the Best Practice method, see Table B-4 in Appendix B.

Acceptance Criteria: Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

NOTE: Only use data associated with acceptable Calibration and QC samples for 625/625.1 compliance samples.

Corrective Action: If any surrogates are outside of the limits, then the following corrective actions must take place (except for dilutions):

* Check all calculations for error.
* Ensure that instrument performance is acceptable.
* Recalculate the data and/or reanalyze the extract if either of the above checks reveals a problem.
* Re-extract and reanalyze the sample or flag the data as "Estimated Concentration" if none of the above resolves the problem.
* If no target analytes are associated with a failed surrogate re-extraction is not required. Write an NCM to identify this.

NOTE: The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare / reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.6.2 If the sample with failed surrogate recoveries was a sample used for an MS/MSD pair and the surrogate recoveries in the MS/MSD are also outside of the control limits, then the sample and the MS and the MSD do not require reanalysis. This phenomenon indicates a possible matrix problem.

9.6.3 If the sample is reanalyzed and the surrogate recoveries in the reanalysis are acceptable, then the problem was within the analyst's control and only the reanalyzed data should be reported. (Unless the reanalysis was outside holding times, in which case reporting both sets of results may be appropriate).

9.6.4 If the reanalysis does confirm the original results, the original analysis is reported and the data flagged as estimated due to matrix effects.

9.6.5 If the sample cannot be re-analyzed for some reason (e.g., not enough volume for re-extraction), then any detected associated compounds in the sample are flagged as estimated due to surrogate failure.

9.7 Internal Standards

9.7.1 The peak areas of the internal standards are monitored in all field samples and QC samples.

Acceptance Criteria: The peak area for each internal standard should be between 50% and 200% of the peak area for the designated midpoint calibration ("ICIS") standard in the

initial calibration. This is typically the 80 ppm Primary (HSL) standard for standard 8270C and the 20 ppm Primary (HSL) standard for LVI.

Corrective Action: If recovery for any internal standard is outside of the control limits, check for calculation errors or instrument problems, and reanalyze the associated samples at a dilution for the compounds quantified against the internal standard that was outside of control limits.

9.7.2 The retention times of the internal standards are monitored in all field samples and QC samples. See Table 15 for DoD/DOE QSM requirements.

Acceptance Criteria: The retention time for any internal standard must be within ± 0.5 minutes of the last continuing calibration standard.

Corrective Action: If the retention time of any internal standard is greater than ± 0.5 minutes the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed if the retention time is outside of limits is required. If the retention time of any internal standard is between ± 0.1 minutes and ± 0.5 minutes, from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP # DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

Samples are prepared according to the appropropriate organic preparation SOPs, listed in Section 2.4.

10.4 Sample Analysis Procedure

10.4.1 Calibrate the instrument as described in Section 11. Depending on the target compounds required by the client, it may be necessary to use more than one set of calibration standards.

10.4.2 All samples must be analyzed using the same instrument conditions as the associated initial calibration and the daily continuing calibration verification (CCV) standard. Extracts are allowed to warm to room temperature before aliquotting. Excess extract is returned to the 0°C to 6°C refrigerator for storage.

10.4.3 Internal Standard

Add internal standard to an aliquot of the extract to result in a 40 ng/µL concentration (for example, 20 µL of internal standard solution at 400 µg/mL in 200 µL of extract). Mix thoroughly before injection into the instrument.

10.4.4 Inject the aliquot into the GC/MS system using the same injection technique as used for the standards.

10.4.4.1 For standard volume extracts the injection volume is 0.5 µL.

10.4.4.2 For LVI, the injection volume is 2.5 µL.

10.4.5 Qualitative identification of each analyte in the extract is evaluated by the data system using the criteria listed in Section 12.2. The data system will determine the concentration of each analyte using calculations equivalent to those in Section 12. Quantitation is based on the initial calibration, not the continuing calibration verification.

10.4.6 Identified compounds are reviewed for proper integration. Manual integrations are performed if necessary and are documented by the analyst (see DV-QA-011P, *Acceptable Manual Integration Practices*) or automatically by the data system. The minimum documentation required includes the original data system peak integration, a similarly scaled hard copy showing the manual integration, the reason for the manual integration, analyst initials and date. All manual integrations must be reviewed by a second level reviewer.

10.4.7 Library searches of peaks present in the chromatogram that are not target compounds, i.e., Tentatively Identified Compounds (TIC), may be performed if required by the client. They are evaluated using the criteria in Section 12.3.

10.5 Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a new aliquot of the extract is diluted with methylene chloride and prepared (IS added to the diluted aliquot) and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below approximately 50% of the calibration range and the matrix allows for analysis at a lesser dilution, the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

10.5.1 Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than the height of the internal standards, or if individual non-target peaks are significantly less than two times the height of the internal standards, the sample should be reanalyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgment. For example, samples containing organic acids may need to be analyzed at a higher dilution to avoid destroying the column.

10.5.2 Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will be reported only at client request.

10.6 Perform all qualitative and quantitative measurements. When the extracts are not being used for analyses, refrigerate them between 0°C to 6°C, protected from light, in screw cap vials equipped with unpierced Teflon lined septa.

10.7 Retention Time Criteria for Samples

10.7.1 If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must

be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required. See Table 15 for DoD/DOE program specific requirements.

10.7.2 If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

10.8 Percent Moisture

Analytical results may be reported as dry or wet weight, as required by the client. Percent moisture must be determined if results will be reported as dry weight. Refer to SOP DV-WC-0023 for determination of percent moisture.

10.9 Data Review

10.9.1 All data are reviewed by the analyst and documented on the checklists, GCMS-Semivolatile Data Review Checklist-CCV and Tune and GCMS-Semivolatile Data Review Checklist-Samples and QC (Form DV-F-0008). See SOP DV-QA-0020, DV-WI-0025 and SOP DV-QA-011P.

10.9.2 The data package and review checklist are submitted to a peer reviewer (or supervisor) for the level 2 review. All manual integrations must be evaluated by the peer reviewer. The level 2 review is documented on the review checklist initiated at the level 1 review. See SOP DV-QA-0020, DV-WI-0025 and SOP DV-QA-011P.

10.9.3 Initial calibrations are subject to a separate two-level review that is documented on a checklist, GC/MS Semivolatile Initial Calibration Data Review Checklist Form DV-F-0007). See SOP DV-QA-0020 and SOP DV-QA-011P.

10.10 Troubleshooting Guide

10.10.1 Daily Instrument Maintenance

In addition to the checks listed in Appendix C, the following daily maintenance should be performed.

- Clip column as necessary.
- Install new or cleaned injection port liner as necessary.
- Install new septum as necessary.
- Install new or cleaned gold seal and washer as necessary.
- Perform mass calibration as necessary.

10.10.2 Major Maintenance

A new initial calibration is necessary following certain maintenance procedures. These maintenance procedures include changing the column, cleaning the repeller, cleaning the source, replacing the multiplier, and replacing the “top board” or RF-related electronics. Refer to the manufacturer's manual for specific guidance.

11.0 Calibration

11.1 Summary

The instrument is tuned for DFTPP, calibrated initially with a minimum of a five levels, and verified each 12-hour shift with one or more continuing calibration standard(s). The number of continuing calibration standards depends upon the target compounds of interest in the analytical batch. Recommended instrument conditions for both the standard analysis and LVI are listed in Table 4.

11.2 All standards and extracts are allowed to warm to room temperature before injecting.

11.3 Instrument Tuning

11.3.1 At the beginning of every twelve-hour shift when analyses are to be performed, the GC/MS system must be checked to see if the acceptance criteria are achieved for DFTPP (decafluorotriphenylphosphine). See Table 5.

11.3.2 Inject 25 ng of the GC/MS tuning standard (Section 7.8) into the GC/MS system. Generate the automatically background-corrected mass spectra of DFTPP from Chrom or other quantitation software and confirm that all the key m/z criteria in Table 5 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed.

11.3.3 The GC/MS tuning standard should also be used to evaluate the inertness of the chromatographic system. The acceptance criteria for the peak tailing factor for benzidine is <3.0 and pentachlorophenol is <5.0. DDT breakdown must be <20%. Refer to Section 12 for the appropriate calculations. See Table 15 for DoD/DOE QSM criteria. See Appendix A for 625/625.1 requirements. If criteria are not met, perform instrument and column maintenance.

11.4 Initial Calibration

11.4.1 Detailed information regarding calibration models and calculations can be found in Corporate SOP CA-Q-P-003, *Calibration Curves and Selection of Calibration Points* and under the public folder, Arizona Calibration Training.

11.4.2 Internal Standard (IS) Calibration Procedure: Internal standards are listed in Table 6. Use the base peak m/z as the primary m/z for quantitation of the standards. If interferences are noted, use one of the next two most intense masses for quantitation.

11.4.3 Compounds are typically assigned to the IS with the closest retention time. IS assignments by compound are listed in Tables 6-8.

11.4.4 A minimum of seven calibration standards are typically prepared, however the number of calibration points may vary. A minimum of five concentration levels are required for each parameter of interest when average response factors or linear regression curve fits are used. At a minimum, six standards must be used

for a quadratic least-squares calibration. It may also be useful to analyze six or seven calibration levels and use the lower five or six for most analytes and the upper five or six for analytes that have poor response. The lowest point on the curve must be at or below the RL of the target analytes.

11.4.5 Rejection of Calibration Points

11.4.5.1 Generally, it is NOT acceptable to remove points from a calibration. If calibration acceptance criteria are not met, the normal corrective action is to examine conditions such as instrument maintenance and accuracy of calibration standards. Any problems must be fixed and documented in the run log or maintenance log. Then the calibration standard(s) must be reanalyzed.

11.4.5.2 If no problems are found or there is documented evidence of a problem with a calibration point (e.g., obvious misinjection explained in the run log), then one point might be rejected, but only if all of the following conditions are met:

* The rejected point is the highest or lowest on the curve, i.e., the remaining points used for calibration must be contiguous; and
* The lowest remaining calibration point is still at or below the project reporting limit; and
* The highest remaining calibration point defines the upper concentration of the working range, and all samples producing results above this concentration are diluted and reanalyzed; and
* The calibration must still have the minimum number of calibration levels required by the method, i.e., five levels for calibrations modeled with average response factors or linear regressions, or six levels for second-order curve fits.

11.4.6 Internal Standards

Add the internal standard mixture to each calibration standard, resulting in a 40 ng/µL final concentration. (For example, if the volume of the calibration standard used is 0.5 mL, add 50 µL of the 400 µg/mL internal standard).

11.4.7 The concentrations of all analytes in the calibration standards are listed in Tables 10, 11 and 12 for the standard analysis and Tables LVI-1, LVI-2 and LVI-3 for LVI. For the Best Practice method, see Table B-2 in Appendix B.

11.4.8 Analyze each calibration standard and tabulate the area of the primary characteristic m/z against the concentration for each compound and internal standard. Calculate the response factors (RF), average response factors, and the percent RSD of the response factors for each compound using the equations in Section 12. Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.10 and 11.4.11, are met. No sample analysis may be performed unless these criteria are met.

11.4.9 Analysis for hexachlorophene is only performed using a single standard run in

the same analytical batch as the sample(s). This provides only semi-quantitative analysis. For quantitative analysis, water samples may be analyzed by LCMSMS, Method 8321A, SOP DV-LC-0022.

11.4.10 Calibration Check Compounds (CCCs)

The %RSD of the response factors for each CCC in the initial calibration must be less than 30% for the initial calibration to be considered valid. This criterion must be met before sample analysis begins. Problems similar to those listed under SPCCs could affect this check.

11.4.10.1 If none of the CCCs are required analytes, then project-specific calibration specifications (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.4.10.2 CCC Compounds:

Phenol
Acenaphthene
1,4-Dichlorobenzene
N-Nitrosodiphenylamine (as Diphenylamine)
2-Nitrophenol
Pentachlorophenol
2,4-Dichlorophenol
Fluoranthene
Hexachlorobutadiene
Di-n-octylphthalate
4-Chloro-3-methylphenol
Benzo(a)pyrene
2,4,6-Trichlorophenol

11.4.11 System Performance Check Compounds (SPCCs)

The minimum average RF for semivolatile SPCCs is 0.050. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

11.4.11.1 SPCC Compounds:

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

11.4.12 If the %RSD of any target analyte is 15% or less, then average relative response factor may be used for quantitation of that analyte.

11.4.12.1 If the %RSD in the initial calibration is > 15%, then calibration using a

curve fit must be used. Linear or quadratic curve fits may be used. If it appears that substantially better accuracy would be obtained using quantitation from a curve fit, then the appropriate curve should be used for quantitation. Use of 1/Concentration or $1/(\text{Concentration})^2$ weighting is recommended to improve the accuracy of quantitation at the low end of the curve. The analyst should consider instrument maintenance to improve the linearity of response.

If a linear regression equation is used, the correlation coefficient r must be greater than 0.990. The Laboratory information management system, TALS, always controls the correlation for curve fits in terms of r^2 which is defined as the mathematical square of the correlation coefficient, r for a linear regression, in which case $r^2>(0.990)^2 = 0.9801$. For a quadratic equation, r^2 and COD are identical and the requirement is that the value will be >0.990. Use of second-order regression equations may be used on rare occasions. In these cases, the intercept and degree of curvature should be examined to be sure that results will be reliable throughout the working range, and the coefficient of determination (r^2) must be greater than 0.990.

Note: South Carolina does not allow use of second order calibration models.

11.4.12.2 An initial calibration verification (ICV) containing all components from a second source (an alternate vendor or a unique lot from the same vendor) must be analyzed after calibration and prior to analyzing other QC or samples. Acceptance criteria for ICV percent recovery (%R) are dependent on the requirements of the client or project. For example, the DoD/DOE QSM requires 80-120% ICV recovery; but non-DoD projects may only require 65-135% for routinely linear compounds (e.g., 625/625.1/8270C HSL components). Clients generally accept laboratory recoveries of 45-155% for poorly performing compounds (e.g., 8270C AP9, Custom list, Refinery list, di-n-butyl phthalate, or benzaldehyde).

Note: Verify that the CCC and SPCC criteria, which are specified in Sections 11.4.10 and 11.4.11, are met. No sample analysis may be performed unless these criteria are met.

Note: DoD/DOE QSM requirements are presented in Table 15.

11.4.13 Weighting of Calibration Data Points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points. 1/Concentration or $1/(\text{Concentration})^2$ weighting (often called 1/x or $1/x^2$

weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

11.4.14 If time remains in the 12-hour period initiated by the DFTPP injection immediately preceding the initial calibration, samples may be analyzed following the initial calibration. Otherwise, proceed to continuing calibration, Section 11.5.

NOTE: Quantitation is performed using the calibration curve or average response factor from the initial curve, not the continuing calibration.

11.5 Continuing Calibration Verification (CCV)

11.5.1 At the start of each 12-hour analysis period, the GC/MS tuning standard must be analyzed. A 25-ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 5.

11.5.2 Following a successful DFTPP analysis, the continuing calibration verification (CCV) standard(s) are analyzed. The standard(s) must contain all semivolatile analytes, including all required surrogates. A mid-level calibration standard is used for the CCV. The standard concentrations used for the CCV are:

Standard ID	Concentration (µg/mL)
Primary (HSL) Standard	80
Appendix 9 Standard (AP9)	80
Refinery (REF)	80
Custom (CUST)	80
Phenyl Compounds (PHE)	80
Dinitroxylenes (DNX)	80
LVI HSL	20
LVI AP9	20
LVI REF	20

11.5.3 The following criteria must be met for the CCV to be acceptable:

11.5.3.1 The SPCC compounds must have a response factor $\geq$ 0.050.

11.5.3.2 The percent difference or drift (%D) of the concentration of the CCC compounds must be $\leq$ 20%. (See Section 12.8 for calculation.)

11.5.3.3 For compounds of interest, reliably performing compounds (see Table 14, List 1 Reliably Performing Compounds) should have a %D $\leq$ 35%. Poorly performing compounds (see Table 15, List 2 Poorly Performing Compounds) should have a %D $\leq$ 50%. Up to 6 target analytes from the entire set of target analytes are allowed to have %D values greater than the applicable limit. Any compound of interest that does not meet the applicable criteria must be documented in a NCM.

11.5.3.4 The internal standard (IS) response of the CCV must be within 50 - 200% of the response in the ICIS of the initial calibration.

11.5.3.5 If any internal standard (IS) retention time in the CCV changes by more than 30 seconds from that of the same level of the corresponding initial calibration, the chromatographic system must be inspected for malfunctions and corrections made as required.

11.5.3.6 If none of the CCCs are required analytes, project-specific calibration requirements (which may include the use of the CCCs listed in Section 11.4.10.2) must be implemented with concurrence from the client.

11.5.4 Once the above criteria have been met, sample analysis may begin. Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs. Analysis may proceed until 12 hours from the injection of the DFTPP have passed. (A sample injected less than or equal to 12 hours after the DFTPP is acceptable.)

NOTE: Some states (like Arizona and South Carolina) have special requirements. Please refer to the posted QAS.

11.5.5 For DoD/QSM 5.0, a closing CCV (CCVC) must be analyzed. If multiple CCVs are used, the first of the CCVCs must be injected prior to the expiration of the 12-hour clock for the analytical batch. See Table 15 for acceptance criteria and corrective actions.

12.0 Calculations / Data Reduction

12.1 Detailed calibration equations can be found in the corporate SOP CA-Q-P-003 *Calibration Curves and Selection of Calibration Points* and under the folder, Arizona Calibration Training.

12.2 Qualitative Identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NBS library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. See Tables 6 and 7 for characteristic ions and approximate retention time order in the primary (HSL) and Appendix 9 standards. Table 8 provides characteristic ions for non-routine compounds.

NOTE: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.

12.2.1 The retention time for the component in the sample must compare to within ± 30 seconds of the retention time of the component in the standard (or other project specific requirement such as DoD/DOE QSM, Table 15). For reference, the

standard must be run within the same twelve hours as the sample unless otherwise directed.

12.2.2 All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.

12.2.3 The characteristic ions of a compound must maximize in the same scan or within one scan of each other.

12.2.4 The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20% and 80%.)

12.2.5 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst the identification is correct, the analyst shall report that identification and proceed with quantitation.

12.3 Tentative Identification

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample spectra with the nearest library searches shall the mass spectral interpretation specialist assign a tentative identification. Following are guidelines for making tentative identification:

12.3.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

12.3.2 The relative intensities of the major ions should agree to within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance should be between 30% and 70%.)

12.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.3.4 Ions present in the sample spectrum, but not in the reference spectrum, should be reviewed for possible background contamination or the presence of co-eluting compounds.

12.3.5 Ions present in the reference spectrum, but not in the sample spectrum, should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

12.3.6 Automatic background subtraction can severely distort spectra from samples with unresolved hydrocarbons.

12.4 Isomers with identical mass spectra and close elution times pose problems for definitive identification. The following compounds, listed in elution order, fall into this category.

aniline and bis(2-chloroethyl) ether
N-nitrosodiphenylamine and diphenylamine
1,3-dichlorobenzene, 1,4-dichlorobenzene, and 1,2-dichlorobenzene
benzyl alcohol, 2-methylphenol, 3-methylphenol, and 4-methylphenol
(Note: 3-methylphenol and 4-methylphenol co-elute)
2- methylnaphthalene and 1-methylnaphthalene
2,4-dimethylphenol and 3,5-dimethylphenol
2,4,6-trichlorophenol and 2,4,5-trichlorophenol
phenanthrene and anthracene
fluoranthene and pyrene
benzo(a)anthracene and chrysene
benzo(e)pyrene and benzo(a)pyrene
bis(2-ethylhexyl)phthalate and di-n-octyl phthalate
benzo(b) and benzo(k)fluoranthene
indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene
safrole and 1-chloronaphthalene
1-naphthylamine and 2-naphthylamine
1-chloronaphthalene and 2-chloronaphthalene
2,4,6-tribromophenol and 2,4,5-tribromophenol

Identification of these compounds requires both experience and extra precautions on the part of the analyst. Specifically, the analyst must more closely scrutinize the comparison of retention times between the unknown and the calibration standard. The analyst must also check that peak resolution is sufficient (valley height of less than 25% of the sum of the two peak heights).

12.5 A second category of problem compounds consist of the poor responders or compounds that chromatograph poorly. The integrations for these types of compounds should be checked manually. The following compounds are included in this category:

Benzoic acid
Chloroanilines
Nitroanilines
2,4-Dinitrophenol
4-Nitrophenol
Pentachlorophenol
3,3'-Dichlorobenzidine
Benzyl alcohol
4,6-Dinitro-2-methylphenol
Atrazine
Famphur
Benzidine

12.6 Relative Response Factor Calculation

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound being measured
A_{is} = Area of the characteristic ion for the specific internal standard
C_x = Concentration of the compound being measured (µg/L)
C_{is} = Concentration of the specific internal standard (µg/L)

12.7 Calculating the Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{\overline{RF}} \times 100\%$$ Where:

$\overline{RF}$ = Mean of RFs from the initial calibration for a compound
SD = Standard deviation for the mean RF from the initial calibration for a compound

$$SD = \sqrt{\frac{\sum_{i=1}^{n}\left(RF_i - \overline{RF}\right)^2}{n-1}}$$

RF_i = RF for each of the calibration levels
n = Number of RF values

12.8 Calculating the Continuing Calibration Percent Drift

$$\%Drift = \frac{C_{found} - C_{actual}}{C_{actual}} \times 100\%$$

Where:

C_{actual} = Known concentration in standard
C_{found} = Measured concentration using selected quantitation method

12.9 Calculating the Concentration in the Extract

The concentration of each identified analyte and surrogate in the extract is calculated from the average RF, or linear or quadratic curve fitted to the initial calibration points. Start with the simplest model, i.e., a straight line through the origin and progress through the other options until the calibration acceptance criteria are met.

12.9.1 Average Response Factor Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is ≤15%, the average response factor from the initial calibration may be used for quantitation. Average response factor is calculated as follows:

$$C_{ex} = \frac{R_x C_{is}}{R_{is}\overline{RF}}$$

Where:

C_{ex} = Concentration in the extract, µg/mL
R_x = Response for the analyte
R_{is} = Response for the internal standard
C_{is} = Concentration of the internal standard

$\overline{RF}$ = Average response factor

12.9.2 Linear Fit Calibration

If the RSD of the response factors for each compound of interest in the initial calibration is >15%, the linear fit calibration may be used for quantitation. To calculate the concentration in an unknown sample extract, the regression equation is solved for:

$$C_{ex} = \frac{\left[\frac{R_x C_{is}}{R_{is}} - b\right]}{m_1}$$

Where:

C_{ex} = Extract analyte concentration, µg/L
R_x = Response for analyte
R_{is} = Response for internal standard
C_{is} = Concentration of internal standard
b = y – Intercept
m_1 = Slope

12.9.3 Quadratic Fit Calibration

When the instrument response does not follow a linear model over a sufficiently wide working range, or when the previously described calibration approaches fail acceptance criteria, a non-linear, second-order calibration model may be employed. To calculate the concentration in an unknown sample extract, the roots of the quadratic equation are solved for:

$$C_{ex} = \frac{-m_1 \pm \sqrt{(m_1)^2 - 4(m_2)\left(b - \frac{R_x C_{is}}{R_{is}}\right)}}{2m_2}$$

Where:

C_{ex} = Extract analyte concentration, µg/L
R_x = Response for analyte
R_{is} = Response for internal standard
C_{is} = Concentration of internal standard
m_2 = Curvature
m_1 = Slope
b = y - Intercept

12.10 Calculating the Concentration in the Sample

12.10.1 Calculation for Aqueous Samples

$$\text{Concentration, } \mu\text{g / L} = \frac{C_{ex}V_t}{V_o}$$

Where:

C_{ex} = Concentration in the extract
V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL.)
V_o = Volume of the sample that was extracted (mL)

12.10.2 Calculation for Sediment, Soil, Sludge, and Waste Samples

Results for sediments, sludges, and soils are usually calculated on a dry-weight basis, and for waste, on a wet-weight basis.

$$\text{Concentration, } \mu\text{g/kg} = \frac{C_{ex}V_t}{W_sD}$$

Where:

C_{ex} = Concentration in the extract
V_t = Volume of total extract in µL, taking into account dilutions (i.e., a 1-to-10 dilution of a 1-mL extract will mean that V_t = 10,000 µL.)
W_s = Weight of sample extracted or diluted in grams
D = (100 - % moisture in sample)/100, for a dry-weight basis or 1 for a wet-weight basis

12.11 MS/MSD Percent Recovery Calculation

$$\text{Matrix Spike Recovery} = \frac{S_{SR} - S_R}{S_A} \times 100\%$$

Where:

S_{SR} = Spike sample result
S_R = Sample result
S_A = Spike added

12.12 Calculating the Relative Percent Difference (RPD) MS/MSD Pair

$$RPD = \frac{|MS_R - MSD_R|}{1/2(MS_R + MSD_R)} \times 100$$

Where:

RPD = Relative percent difference
MS_R = Matrix spike result
MSD_R = Matrix spike duplicate result

12.13 Calculation of TICs

The calculation of TICs (tentatively identified compounds) is identical to the RF calculation (12.6) with the following exceptions:

A_x = Area of the total ion chromatogram for the compound being measured
A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference
RF = 1

12.14 Calculating Percent DDT Breakdown

$$\%\ \text{DDT breakdown} = \frac{\text{DDEarea} + \text{DDDarea}}{\text{DDTarea} + \text{DDEarea} + \text{DDDarea}}$$

The areas for the m/z 235 ion are used for this calculation.

12.15 Calculating the Peak Tailing Factor

$$\text{Tailing Factor} = \frac{BC}{AB}$$

Where:

Peak width (AC) is measured at 10% peak height, and divided into two line segments at the peak centroid, so that

AC = AB + BC, with
AB = left-hand segment
BC = right-hand segment

13.0 <u>Method Performance</u>

13.1 Method Detection Limit Study (MDL)

13.1.1 The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in CA-Q-S-006. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

13.1.2 Non-Routine Analytes

For non-routine analytes, an MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration should include the analysis of an extracted standard at the reporting limit and a single point calibration.

13.2 MDLV and LOQV

13.2.1 MDLVs and LOQVs must be performed on blank matrix as defined for the method blank (Section 9.3), spiked with the appropriate analytes at the programmatically required spike amounts.

13.2.2 MDLVs are analyzed for compounds on the DOD certificate and those required for Texas TRPP on each instrument each quarter. For DOD the spike requirement is 2-4X the MDL and for Texas it is 1-4X the MDL. There are no recovery requirements for MDLV. The MDLs for all other compounds are verified at least annually

13.2.3 LOQVs (aka LLOQV) are analyzed for compounds on the DOD certificate on each instrument each quarter. The spiking requirement is 1-2X the RL and must pass percent recovery limits. For all compounds analyzed by Method 8260, LOQVs must be performed annually for compliance with Method 8000D, SW-846 Update V.

13.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

13.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

13.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

13.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

13.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

13.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

14.0 Pollution Control

14.1 Standards and reagents should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards and reagents requiring disposal.

14.2 The LVI method utilized smaller sample sizes and extraction volumes resulting in a decrease in the amount of waste generated for the analysis of water samples.

15.0 Waste Management

15.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, "Waste Management and Pollution Prevention", of the Environmental Health and Safety Manual, and DV-HS-001P, "Waste Management Program."

15.2 The following waste streams are produced when this method is carried out:

15.2.1 Expired Chemicals/Reagents/Standards – Contact Waste Coordinator

15.2.2 Methylene Chloride – B

15.2.3 Flammable Solvent – Waste Stream C

15.2.4 Used vials – Waste Stream A

NOTE: Radioactive, mixed waste, and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

16.0 References / Cross-References

16.1 40 CFR Part 136, Appendix A (Method 624, Method 603) and MUR update on August 28, 2018.

16.2 SW846, Test Methods for Evaluating Solid Waste, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

16.2.1 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Capillary Column Technique, Update III, December

1996.

16.2.2 Method 8000B, Determinative Chromatographic Separations, Revision 2, December 1996.

16.2.3 Method 8000C, Determinative Chromatographic Separations, Revision 3, March 2003.

16.2.4 Method 8000D, Determinative Chromatographic Separations, Revision 4, July 2014.

16.2.5 Method 3510C, Separatory Funnel Liquid-Liquid Extraction, Revision 3, December 1996.

16.2.6 Method 3520C, Continuous Liquid-Liquid Extraction, Revision 3, December 1996.

16.2.7 Method 3546, Microwave Extraction, Revision 0, February 2007.

16.2.8 Method 3550B, Ultrasonic Extraction, Revision 2, December 1996.

16.2.9 Method 3550C, Ultrasonic Extraction, Revision 3, February 2007.

16.2.10 Method 3580, Waste Dilution, Revision 1, July 1992.

16.2.11 Method 3620C, Florisil Cleanup, Revision 3, February 2007.

16.2.12 Method 3660B, Sulfur Cleanup, Revision 2, December 1996.

16.2.13 Method 3665A, Sulfuric Acid/Permanganate Cleanup, Revision 1, December 1996.

16.3 40 CFR, part 136, Appendix A, "Base/Neutrals and Acids", Method 625/625.1.

17.0 Method Modifications:

17.1 Modifications from Reference Method

17.1.1 This SOP evaluates the hardware tune based on injection of 25 ng DFTPP compared to the 50 ng DFTPP stated in Method 8270C and Method 625/625.1.

17.1.2 This procedure does not use the relative retention time units specified in the reference method since some data systems do not have this capability. Instead, retention time windows of ± 0.5 minute from the preceding continuing calibration are used for all internal standards. A comparison of retention time shifts within these windows shows that they are well within the required relative retention time window of 0.06 specified in method 8270C.

17.1.3 The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.4 This procedure includes the option for weighted linear regression curves using 1/(concentration)^2 weighting factors. With an unweighted linear regression, points at the lower end of the calibration curve have less weight in determining the curve than points at the high concentration end of the curve. For this reason, inverse weighting of the linear function is recommended to optimize the accuracy at low concentrations.

Note that the August 7, 1998 EPA memorandum "Clarification Regarding Use of SW-846 Methods", Attachment 2, Page 9, includes the statement "The Agency further recommends the use of this form of weighted regression over the use of an unweighted regression."

17.1.5 This procedure uses specific criteria to quantifiably evaluate the tailing factors of benzidine and pentachlorophenol (for calculations see section 12.15). Method 8270C only specifies that no peak tailing should be visible. To avoid subjectivity, no visible tailing is defined as a tailing factor of <3.0 for benzidine and <5.0 for pentachlorophenol.

17.1.6 This procedure uses final surrogate concentrations of 100 ng/µL for the acid surrogates and 100 ng/µL for base/neutral surrogates. Method 8270C specifies that 1-2 uL injections contain 200 ng of acid surrogates and 100 ng of the base/neutral surrogates (assuming 100% recovery). Based on a 0.5 uL injection, this procedure uses concentrations approximately 0.5 to 3 times those mentioned in Method 8270C.

17.1.7 The Large Volume Injection technique utilizes modifications in the source methods regarding both sample size and injection volume. These modifications are explicitly allowed by EPA. SW-846, Chapter Two lists as acceptable variations adjustment to both the sizes of sample prepared and injection volumes. See TestAmerica White Paper No. CA-Q-W-010, "Large Volume Injection (LVI) Technique for Organic Preparation and Analysis Methods" for detailed discussions. Reporting limits remain the same for this technique.

17.1.8 The laboratory uses a % drift or % difference for poor performing compounds of 50% compared to the 35% in the method for evaluation of the CCV. CCV control charts will be periodically reviewed to assess the reliability of this approach.

18.0 Attachments

Table 1. TestAmerica Primary Standard and Standard Reporting Limits
Table 2. TestAmerica Appendix IX Standard Reporting Limits
Table 3. TestAmerica Non-Routine Compound Reporting Limits
Table 4. Suggested Instrument Conditions
Table 5. DFTPP Key Ions and Ion Abundance Criteria
Table 6. Characteristic Ions, Primary Standard (in approximate retention time order)
Table 7. Characteristic Ions, Appendix IX Standard (in approximate retention time order)
Table 8. Characteristic Ions, Non- Routine Compounds
Table 9. 8270C Surrogate Compounds
Table 10. Calibration Levels, Primary (HSL) Standard, µg/mL
Table 11. Calibration Levels, Appendix IX Standard, µg/mL
Table 12. Calibration Levels, Non-routine Compounds, µg/mL

Table 13. List 1 – Reliably Performing Compounds
Table 14. List 2 – Poorly Performing Compounds.
Table 15. DOD QSM 5.0 GCMS Method Requirements
Table LVI-1 LVI Calibration Levels, Primary (HSL) Standard, µg/mL
Table LVI-2 LVI Calibration Levels, Appendix IX Standard, µg/mL
Table LVI-3 LVI Calibration Levels, Non-routine Compounds, µg/mL

APPENDIX A. Modifications Required for Analysis of Wastewater Following Method 625/625.1

Table A-1. TestAmerica Method 625/625.1 Standard Reporting List and Reporting Limits
Table A-2. Initial Demonstration Recovery and Precision Limits for Method 625/625.1

APPENDIX B. Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (8270BP)

Table B-1. TestAmerica Method 8270BP Standard Reporting Limits
Table B-2. Method 8270BP Calibration Levels
Table B-3. Method 8270BP LCS Spike Concentrations
Table B-4. 8270BP Surrogate Compounds
Table B-5. 8270BP Internal Standard Compounds
Table B-6. Suggested Instrument Conditions for 8270BP
Table B-7. Internal Standard and Surrogate Association for 8270BP Base-Only Extraction

APPENDIX C. Instrument Maintenance Schedules - Mass Spectrometer & Gas Chromatograph

19.0 Revision History

- Revision 16, dated 4 March 2020
  - Annual Review
  - Updated Copyright Information

- Revision 15, dated 18 January 2019
  - Added section 3.9 defining LLOQ checks.
  - Added section 13.2 regarding MDLV/LOQV criteria.
  - Added section 16.2.4 referencing method 8000D.

- Revision 14, dated 31 March 2018
  - Added notes in QC sections for 625.1 analysis.
  - Updated References for MUR method 5 update.

- Revision 13, dated 30 September 2017
  - Annual Review

- Revision 12, dated 31 August 2016
  - Removed famphur from Section 1.2.8 and added statement that hexachlorophene is only presented as semi-quantitative result

- Removed references to DV-OP-0010 and SW-846 3540C; lab does not currently have the SW-846 3540C as a soil prep associated with Method 8270C
- Revised Section 2.6 to reflect current practice
- Added clarification in Section 3.3.2 that when DoD samples are the only samples in the batch an LCSD is not run in the absence of field assigned MS/MSD
- Added new section 6.10 to describe injection port liners used in method
- Added statement to section 7.2 that equivalent standards to those named in the succeeding sections could be used if the named standards were not available
- Added statement to section 7.11 that standards are warmed to RT prior to injection
- Added notes and statements that DoD/DOE QSM criteria are found in Table 15 in Sections: 9.1.4, 9.7.2, 10.7.1, 11.3.3, 11.4.12.2, 11.5.5, 12.2.1
- Added statement to Section 9.4 regarding when to use full analyte spikes
- Revised Section 9.5 to reflect current policy for evaluation of MS/MSD
- Added bullet to Section 9.6.1 corrective action regarding surrogate not assigned to a target compound
- Removed prep SOP numbers/titles from Section 10.3 and referenced Section 2.4 where the same information is listed.
- Added statement that samples are analyzed under the same conditions as the associated ICAL (Section 10.4.2)
- Removed statement regarding L4 documentation in Section 10.4.6. Current practice is compliant with DoD/DOE QSM requirements.
- Updated data review checklist names in Section 10.9 to reflect current practice.
- Added corrective action if peak tailing and breakdown criteria not met (Section 11.3.3).
- Added reference to tables for IS assignments (Section 11.4.3)
- Clarified relationship between low point of curve and standard RL (Section 11.4.4)
- Added new section 11.4.9 to describe analysis of hexachlorophene and recommendation to use LCMSMS method; renumbered remainder of section
- Aligned requirements for second source standard with DV-QA-0015 in Section 11.4.12.2 and revised section for clarity
- Removed description of V_t when combining extracts after concentration. This is no longer standard practice in the prep lab. (Sections 12.10.1 and 12.10.2)
- Revised Section 13 to reflect current practice.
- Added Section 17.1.8 to address acceptance criteria for poorly performing compounds.
- Revised DFTPP criteria for ion 442 to reflect source method requirements and current practice.
- Revised tables in Appendix B-1 to reflect current practice.
- Removed general references to AFCEE
- Updated section references and formatting and made editorial changes throughout

- Revision 11, dated 30 June 2015
  - Changed corporate SOP reference for calibration curves from CA-Q-S-005 to CA-Q-P-003 to accommodate document revision change issued by Corporate QA (Sections 11.4.1 and 12.1)
  - Revised Section 12.4, isomers with identical mass spectra and close elution times, for consistency with data review checklist
  - Revised language in Section 17.1.4 referencing EPA memo on weighted linear regression to be consistent with other analytical SOPs in use in the laboratory.
  - Revised Titles in Tables 3 and 8 to Non-routine in place of Non-standard for consistency with terminology throughout SOP
  - Moved Table 13 to Appendix A and renumbered as Table A-2. Renumbered existing Tables 14-16 to Tables 13-15.
  - Revised Table 15 to clarify DoD QSM 5 requirements or correct omissions.
  - Added bullet to Appendix A to address precision QC element required by EPAs 2012 MUR to 40 CFR Part 136
  - Edited Appendix B, point 3 to include option for base-only extraction.
  - Added clarification to both Appendix A and Appendix B that the characteristic ions, IS and surrogate assignments are those given in Tables 6-8 except as noted for the base-only extraction in Appendix B.
  - Added Table B-7 for IS and surrogate assignments for base-only extraction
  - Formatting changes throughout

- Revision 10, dated 31 January 2014
  - Removed Section 9.8 referencing work instruction on MUR and added MUR requirements to appropriate sections in SOP
  - Added analyte pairs to section 12.4
    2,4-Dimethylphenol and 3,5-Dimethylphenol
    Benzo(e)pyrene and Benzo(a)pyrene
  - Added Table 16 – DoD QSM 5 requirements for GCMS methods

- Revision 9, dated 31 January 2014
  - Updated calibration standards and their corresponding sublists throughout the SOP (including text, tables and appendices).
    - 1,1-Biphenyl, Acetophenone, 3-Methylphenol, 1,2,4,5-Tetrachlorobenzene, 1,3-Dinitrobenzene and 2,3,4,6-Tetrachlorophenol moved into the HSL sublist
    - 4,4'-Methylene bis(2-chloroaniline), 6-Methylchrysene, Acrylamide, Dibenz(a,h)acridine, and Quinoline moved into the AP9 sublist.
  - Modified the large volume injection (LVI) injection volume to 2.5 µL and internal standard concentration to 40 µg/mL.
  - Formatting and editorial changes throughout.

- Revision 8, dated 31 January 2013
  - Added information to analyze water extracts using Large Volume Injection throughout the SOP
  - Identified surrogate and internal standard associations and standard name for non-standard compounds in Table 8
  - Added surrogate concentrations for LVI extracts in Table 9

- Added data for calibration standards identified as Refinery, Custom, PHE, and Benzaldehyde in Table 12
- Added Tables LVI-1, LVI-2 and LVI-3 (LVI calibration standards)
- Revised sections 9.1, 10.1 and 10.2 to reflect current practice
- Removed AFCEE calibration curve (no longer used)
- Formatting and editorial changes throughout
- Added section 9.8 for 2012 MUR QC requirements

- Revision 7, dated 28 September 2012
  - Added Table 3 TestAmerica Non-Standard Compound Reporting Limits
  - Added Table 8 Characteristic Ions, Non- Standard Compounds
  - Updated references to tables
- Revision 6, dated 9 January 2012
  - Source method review
  - Clarified procedural details in section 10, 11 and 12 including expanding ICV and CCV acceptance criteria for various programs
  - Revised Section 12
  - Updated method modifications section
  - Expanded Table 6 to include IS and Surrogate compound assignments for all target analytes
  - Removed Tables 7 and 8 and referenced Organic Prep SOPs and TALS reagent module for composition and concentration of LCS solutions. Renumbered remaining tables
  - Removed Table A-2 and inserted reference to organic prep SOPs and TALS reagent module for composition and concentration of LCS and MS solutions for Method 625
- Revision 5.3, dated 31 May 2011
  - Annual review
  - Reorganized and updated Section 9
  - Added Section 10.8 on data review
  - Updated sections for references to current LIMS system and current practices
  - Expanded reference section to include all prep methods and Method 8000
  - Updated Section 17
  - Formatting and grammatical changes throughout

Earlier revision histories have been archived and are available upon request.

Table 1.
TestAmerica Primary Standard and Standard Reporting Limits

		Standard Reporting Limits	
Analytes	**CAS Number**	**Aqueous (µg/L)**	**Low Soil/Sediment (µg/kg)**
Pyridine	110-86-1	20	660
N-Nitrosodimethylamine	62-75-9	10	330
Aniline	62-53-3	10	330
Phenol	108-95-2	10	330
Bis(2-chloroethyl)ether	111-44-4	10	330
2-Chlorophenol	95-57-8	10	330
1,3-Dichlorobenzene	541-73-1	10	330
1,4-Dichlorobenzene	106-46-7	4	330
Benzyl alcohol	100-51-6	10	330
1,2-Dichlorobenzene	95-50-1	4	330
2-Methylphenol	95-48-7	10	330
2,2'-oxybis(1-chloropropane)	108-60-1	10	330
4-Methylphenol	106-44-5	10	330
N-Nitroso-di-n-propylamine	621-64-7	10	330
Hexachloroethane	67-72-1	10	330
Nitrobenzene	98-95-3	10	330
Isophorone	78-59-1	10	330
2-Nitrophenol	88-75-5	10	330
2,4-Dimethylphenol	105-67-9	10	330
Benzoic acid	65-85-0	25	1600
Bis(2-chloroethoxy)methane	111-91-1	10	330
2,4-Dichlorophenol	120-83-2	10	330
1,2,4-Trichlorobenzene	120-82-1	4	330
Naphthalene	91-20-3	10	330
4-Chloroaniline	106-47-8	10	330
Hexachlorobutadiene	87-68-3	10	330
1,2,4,5-Tetrachlorobenzene	95-94-3	10	330
4-Chloro-3-methylphenol	59-50-7	10	330
2-Methylnaphthalene	91-57-6	10	330
Hexachlorocyclopentadiene	77-47-4	50	1600
2,4,6-Trichlorophenol	88-06-2	10	330
2,4,5-Trichlorophenol	95-95-4	10	330
2-Chloronaphthalene	91-58-7	4	330
2-Nitroaniline	88-74-4	10	1600
Dimethyl phthalate	131-11-3	4	330
1,3-Dinitrobenzene	99-65-0	10	330
Acenaphthylene	208-96-8	4	330
3-Nitroaniline	99-09-2	10	1600
Acenaphthene	83-32-9	4	330
2,4-Dinitrophenol	51-28-5	30	1600
4-Nitrophenol	100-02-7	10	1600
Dibenzofuran	132-64-9	4	330
2,3,4,6-Tetrachlorophenol	58-90-2	50	1600
2,4-Dinitrotoluene	121-14-2	10	330
2,6-Dinitrotoluene	606-20-2	10	330

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Table 1 (continued)
TestAmerica Primary Standard and Standard Reporting Limits

Analytes	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Diethyl phthalate	84-66-2	4	330
4-Chlorophenyl phenyl ether	7005-72-3	10	330
Fluorene	86-73-7	4	330
4-Nitroaniline	100-01-6	10	1600
4,6-Dinitro-2-methylphenol	534-52-1	20	1600
N-Nitrosodiphenylamine	86-30-6	10	330
Azobenzene	103-33-3	10	330
4-Bromophenyl phenyl ether	101-55-3	10	330
Hexachlorobenzene	118-74-1	10	330
Pentachlorophenol	87-86-5	50	1600
Phenanthrene	85-01-8	4	330
Anthracene	120-12-7	4	330
Carbazole	86-74-8	4	330
Di-n-butyl phthalate	84-74-2	4	330
Fluoranthene	206-44-0	4	330
Benzidine	92-87-5	100	3300
Pyrene	129-00-0	10	330
Butyl benzyl phthalate	85-68-7	4	330
3,3'-Dichlorobenzidine	91-94-1	50	1600
Benzo(a)anthracene	56-55-3	4	330
Bis(2-ethylhexyl)phthalate	117-81-7	10	330
4,4-Methylenebis(2-chloroaniline)	101-14-4	100	330
Chrysene	218-01-9	4	330
Di-n-octylphthalate	117-84-0	4	330
Benzo(b)fluoranthene	205-99-2	4	330
Benzo(k)fluoranthene	207-08-9	4	330
Benzo(a)pyrene	50-32-8	4	330
Indeno(1,2,3-cd)pyrene	193-39-5	4	330
Diethyl phthalate	84-66-2	4	660
Dibenz(a,h)anthracene	53-70-3	4	330
Benzo(g,h,i)perylene	191-24-2	4	330
Acetophenone	98-86-2	10	330
3/4-Methylphenol	15831-10-4	10	330
1,4-Dioxane	54841-74-6	20	660

1. The TAL primary standard, or HSL standard is the standard normally used at TAL. Additional standards, such as the Appendix IX standard (AP9), the Refinery standard (REF), the Custom standard (CUST), the Dinitroxylenes standard (DNX), Benzaldehyde standard (BZHD) or the phenyl standard (PHE) may be necessary to include all target analytes required for some clients.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 2.
TestAmerica Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
2-Picoline	109-06-8	20	660
N-Nitrosomethylethylamine	10595-95-6	10	330
Methyl methanesulfonate	66-27-3	10	330
N-Nitrosodiethylamine	55-18-5	10	330
Ethyl methanesulfonate	62-50-0	10	330
Pentachloroethane	76-01-7	50	1600
N-Nitrosopyrrolidine	930-55-2	10	330
N-Nitrosomorpholine	59-89-2	10	330
o-Toluidine	95-53-4	10	660
N-Nitrosopiperidine	100-75-4	10	330
o,o,o-Triethyl-Phosphorothioate	126-68-1	50	1600
a,a-Dimethyl-phenethylamine	122-09-8	50	1600
2,6-Dichlorophenol	87-65-0	10	330
Hexachloropropene	1888-71-7	100	3300
p-Phenylenediamine	106-50-3	100	1600
n-Nitrosodi-n-butylamine	924-16-3	10	330
Safrole	94-59-7	50	1600
Isosafrole	120-58-1	20	660
1,4-Dinitrobenzene	100-25-4	10	330
1,4-Naphthoquinone	130-15-4	50	1600
Pentachlorobenzene	608-93-5	10	330
1-Naphthylamine	134-32-7	10	330
2-Naphthylamine	91-59-8	10	330
5-Nitro-o-toluidine	99-55-8	20	660
Thionazin	297-97-2	10	1600
1,3,5-Trinitrobenzene	99-35-4	50	1600
Sulfotepp	3689-24-5	50	1000
Phorate	298-02-2	50	1600
Phenacetin	62-44-2	20	660
Diallate	2303-16-4	20	660
Dimethoate	60-51-5	20	660
4-Aminobiphenyl	92-67-1	50	1600
Pentachloronitrobenzene	82-68-8	50	1600
Pronamide	23950-58-5	20	660
Disulfoton	298-04-4	50	1600
2-secbutyl-4,6-dinitrophenol (Dinoseb)	88-85-7	10	660
Methyl Parathion	298-00-0	50	1600
1-Chloronaphthalene	90-13-1	10	330
Biphenyl	92-51-3	10	330
4-Nitroquinoline-1-oxide	56-57-5	100	3300
Parathion	56-38-2	50	1600
Methapyrilene	91-80-5	50	1600

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Table 2. (continued)
TestAmerica Appendix IX Standard Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Aramite	140-57-8	20	660
Isodrin	465-73-6	10	330
p-(Dimethylamino)azobenzene	60-11-7	20	660
p-Chlorobenzilate	510-15-6	10	330
3,3'-Dimethylbenzidine	119-93-7	20	660
2-Acetylaminofluorene	53-96-3	100	3300
Dibenz(a,j)acridine	224-42-0	10	660
7,12-Dimethylbenz(a)anthracene	57-97-6	20	660
3-Methylcholanthrene	56-49-5	20	660
Diphenylamine	122-39-4	10	330

1. The Appendix IX standard contains additional analytes required for the Appendix IX list. The TAL primary standard must also be analyzed to include all of the Appendix IX list.
2. May also be analyzed by method 8141, which can achieve lower reporting limits.
3. May also be analyzed by method 8080 or 8081, which can achieve lower reporting limits.

Table 3.
TestAmerica Non-Routine Compound Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
1,1'-Biphenyl	92-52-4	10	330
1,2-Dimethyl-3,4-Dinitrobenzene	603-06-5	5.0	167
1,2-Dimethyl-3,5-Dinitrobenzene	STL01265	5.0	167
1,2-Dimethyl-3,6-Dinitrobenzene	STL01263	5.0	167
1,2-Dimethyl-4,5-Dinitrobenzene	3395-03-7	5.0	167
1,3-Dimethyl-2,4-Dinitrobenzene	610-23-1	5.0	167
1,3-Dimethyl-2,5-Dinitrobenzene	STL01264	5.0	167
1,3-Phenylenediamine	108-45-2	10	660
1,4-Dimethyl-2,3-Dinitrobenzene	711-41-1	5.0	167
1,4-Dimethyl-2,6-Dinitrobenzene	STL01266	5.0	167
1,5-Dimethyl-2,3-Dinitrobenzene	65151-56-6	5.0	167
1,5-Dimethyl-2,4-Dinitrobenzene	616-72-8	5.0	167
1-Methylnaphthalene	90-12-0	4.0	330
2,3-Dichlorobenzenamine	608-27-5	10	330
2,4,6-Tribromophenol	118-79-6	5	330
2,4-Xylidine	95-68-1	10	660
2-Ethoxyethanol	110-80-5	100	3300
3,5-Dimethylphenol	108-68-9	10	330
3-Methylphenol	108-39-4	10	330
4,4-Dichlorobenzil	3457-46-3	10	330
4-Chlorobenzenethiol	106-54-7	10	330
5-Methyl-o-Anisidine	120-71-8	10	660
6-Methylchrysene	1705-85-7	10	330
Acrylamide	79-06-1	200	1600
Alachlor	15972-60-8	20	330
Alpha Methyl Styrene	98-83-9	10	330
Alpha-Terpineol	98-55-5	5.0	330
Atrazine	1912-24-9	10	330
Benzaldehyde	100-52-7	10	330
Benzo(e)pyrene	192-97-2	4	330
Benzyl dichloride	98-87-3	50	2700
Bis(4-Chlorophenyl)disulfide	1142-19-4	10	330
Caprolactam	105-60-2	10	1600
Carbofuran Phenol	1563-38-8	50	2700
Dibenz(a,h)acridine	226-36-8	10	330
Dibenzo(a,e)pyrene	192-65-4	10	1600
Dimethylformamide	68-12-2	50	330
Diphenylsulfone	127-63-9	10	330
Famphur	52-85-7	100	660
Hexachlorophene	70-30-4	1000	33000
Hexadecane	544-76-3	10	330
Indene	95-13-6	10	330
Methyl 4-Chlorophenyl Sulfide	123-09-1	10	330

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Table 3. (continued)
TestAmerica Non-Routine Compound Reporting Limits

Semivolatiles	CAS Number	Standard Reporting Limits	
		Aqueous (µg/L)	Low Soil/Sediment (µg/kg)
Alpha Methyl Styrene	25013-15-4	10	330
n-Decane	124-18-5	10	330
n-Hydroxymethlphthalimide	118-29-6	10	330
o-Anisidine	90-04-0	10	660
Octachlorostyrene	29082-74-4	10	330
p-Chlorophenyl Sulfone	80-07-9	10	330
Perylene	198-55-0	10	1600
Phenyl Disulfide	882-33-7	10	330
Phenyl Sulfide	139-66-2	10	330
Phenylmercaptan	108-98-5	100	3300
Phthalic Acid	88-99-3	400	2500
Phthalic Anhydride	85-44-9	400	2500
Quinoline	91-22-5	50	1600
Toluene Diamine (2,4- + 2,6 – Isomers)	STL00848	1000	1600
Tributyl Phosphate	126-73-8	50	1600
Triethyl Amine	121-44-8	100	2700
Triethyl Phosphate	78-40-0	100	1600
Tris (2,3-dibromopropyl)phosphate	126-72-7	100	6600

Table 4.
Suggested Instrument Conditions

Parameter	Settings	
	Standard Analysis	**Large Volume Injection**
Mass Range:	35 - 500 amu	35-500 amu
Scan Time:	≤ 1 second/scan	≤ 1 second/scan
Initial Column Temperature/Hold Time:	40 °C for 1 minute	55 °C for 1.5 minute
Column Temperature Program:	40 - 325 °C at 25 °C/min	55 - 190 °C at 25 °C/min 190 - 330 °C at 28 °C/min
Final Column Temperature/Hold Time:	325 °C (until at least one minute after benzo(g,h,i)perylene has eluted)	330 °C (until at least one minute after benzo(g,h,i)perylene has eluted)
Injector Temperature:	250 °C	250 °C
Transfer Line Temperature:	290 °C	300 °C
Source Temperature:	According to manufacturer's specifications	According to manufacturer's specifications
Injector:	Split / splitless injection liner; Restek liner #23303.5 or equivalent	Split / splitless injection liner; Restek liner #23303.5 or equivalent
Injection Mode	Split (3:1)	Split (3:1)
Syringe	5 or 10 µL	5 or 10 µL
Sample Volume:	0.5 µl	2.5 µL
Carrier Gas:	Helium at 3.4 mL/min	Helium at 3.4 mL/min

Table 5.
DFTPP Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
51	30 - 60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40 - 60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative abundance
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17 - 23% of mass 442

Table 6.
Characteristic Ions, Primary Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	Surr
1,2,4,5-Tetrachlorobenzene	216	214	218	2	4
1,2,4-Trichlorobenzene	180	182	145	2	4
1,2-Dichlorobenzene	146	148	113	1	4
1,3-Dichlorobenzene	146	148	113	1	4
1,3-Dinitrobenzene	168	50	76	3	2
1,4-Dichlorobenzene	146	148	113	1	4
1,4-Dichlorobenzene-d_4 (Internal Standard)	152	150	115	1	-
1,4-Dioxane	88	58	-	1	3
2,2'-oxybis(1-chloropropane)[1]	45	77	79	1	4
2,3,4,6-Tetrachlorophenol	232	230	131	3	2
2,4,5-Trichlorophenol	196	198	200	3	1
2,4,6-Tribromophenol (Surrogate Standard)	330	332	141	3	1
2,4,6-Trichlorophenol	196	198	200	3	1
2,4-Dichlorophenol	162	164	98	2	3
2,4-Dimethylphenol	107	121	122	2	3
2,4-Dinitrophenol	184	63	154	3	1
2,4-Dinitrotoluene	165	63	89	3	2
2,6-Dinitrotoluene	165	63	89	3	2
2-Chloronaphthalene	162	164	127	3	2
2-Chlorophenol	128	64	130	1	3
2-Fluorobiphenyl (Surrogate Standard)	172	171	170	3	2
2-Fluorophenol (Surrogate Standard)	112	64	63	1	3
2-Methylnaphthalene	142	141	115	2	2
2-Methylphenol	108	107	77	1	3
2-Nitroaniline	65	92	138	3	2
2-Nitrophenol	139	65	109	2	3
3,3'-Dichlorobenzidine	252	254	126	5	2
3/4-Methylphenol	108	107	77	1	3
3-Nitroaniline	138	108	92	3	2
4,6-Dinitro-2-methylphenol	198	105	51	4	1
4-Bromophenylphenylether	248	250	141	4	2
4-Chloro-3-methylphenol	107	144	142	2	1
4-Chloroaniline	127	129	65	2	4
4-Chlorophenylphenylether	204	206	141	3	2
4-Methylphenol	108	107	79	1	3
4-Nitroaniline	138	92	108	3	2
4-Nitrophenol	109	139	65	3	1
Acenaphthene	153	152	154	3	6
Acenaphthene-d_{10} (Internal Standard)	164	162	160	3	-
Acenaphthylene	152	151	153	3	6
Acetophenone	105	77	120	1	3
Aniline	93	66	-	1	3
Anthracene	178	179	176	4	6
Azobenzene	77	182	105	3	2
Benzidine	184	92	185	5	6

Table 6. (continued)
Characteristic Ions, Primary Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	Surr
Benzo(a)anthracene	228	229	226	5	6
Benzo(a)pyrene	252	253	125	6	6
Benzo(b)fluoranthene	252	253	125	6	6
Benzo(g,h,i)perylene	276	138	277	6	6
Benzo(k)fluoranthene	252	253	125	6	6
Benzoic Acid	122	105	77	2	3
Benzyl Alcohol	108	79	77	1	4
Bis(2-chloroethoxy)methane	93	95	123	2	4
Bis(2-chloroethyl)ether	93	63	95	1	4
Bis(2-ethylhexyl)phthalate	149	167	279	5	2
Butylbenzylphthalate	149	91	206	5	2
Carbazole	167	166	139	4	1
Chrysene	228	226	229	5	6
Chrysene-d_{12} (Internal Standard)	240	120	236	5	-
Dibenz(a,h)anthracene	278	139	279	6	2
Dibenzofuran	168	139	84	3	2
Diethylphthalate	149	177	150	3	2
Dimethylphthalate	163	194	164	3	2
Di-n-butylphthalate	149	150	104	4	2
Di-n-octylphthalate	149	167	43	5	2
Famphur	218	93	125	5	6
Fluoranthene	202	101	100	4	6
Fluorene	166	165	167	3	6
Hexachlorobenzene	284	142	249	4	2
Hexachlorobutadiene	225	223	227	2	4
Hexachlorocyclopentadiene	237	235	271	3	2
Hexachloroethane	117	201	199	1	4
Indeno(1,2,3-cd)pyrene	276	138	277	5	6
Isophorone	82	95	138	2	4
Naphthalene	128	129	127	2	6
Naphthalene-d_8 (Internal Standard)	136	68	54	2	-
Nitrobenzene	77	123	65	2	4
Nitrobenzene-d_5 (Surrogate Standard)	82	128	54	2	4
N-Nitrosodimethylamine	74	42	-	1	3
N-Nitroso-di-n-propylamine	70	42	101,130	1	4
N-Nitrosodiphenylamine	169	168	167	4	2
Pentachlorophenol	266	264	268	4	1
Perylene-d_{12} (Internal Standard)	264	260	265	6	-
Phenanthrene	178	179	176	4	6
Phenanthrene-d_{10} (Internal Standard)	188	94	80	4	-
Phenol	94	65	66	1	5
Phenol-d_5 (Surrogate Standard)	99	42	71	1	5
Pyrene	202	101	100	5	6
Pyridine	79	52	-	1	3
Terphenyl-d_{14} (Surrogate Standard)	244	122	212	5	6

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether.

Table 7.
Characteristic Ions, Appendix IX Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	SURR
1,3,5-Trinitrobenzene	213	75	120	4	1
1,4-Dinitrobenzene	168	75	122	3	2
1,4-Naphthoquinone	158	104	102	3	2
1-Naphthylamine	143	115	--	3	2
2,6-Dichlorophenol	162	164	63	2	4
2-Acetylaminofluorene	181	180	223	5	6
2-Naphthylamine	143	115	--	3	2
2-Picoline	93	66	92	1	3
2-sec-butyl-4,6-dinitrophenol (Dinoseb)	211	163	147	4	1
3,3'-Dimethylbenzidine	212	106	--	5	6
3-Methylcholanthrene	268	252	253	6	6
4-Aminobiphenyl	169	168	115	4	1
4-Nitroquinoline-1-oxide	190	128	160	4	6
5-Nitro-o-toluidine	152	77	106	3	2
7,12-Dimethylbenz(a)anthracene	256	241	120	6	6
a,a-Dimethyl-phenethylamine	58	91	--	2	4
Aramite 1	185	319	--	5	6
Aramite 2	185	319	--	5	6
Diallate	86	234	--	4	1
Dibenz(a,j)acridine	279	280	--	6	6
Dimethoate	87	93	125	4	1
Disulfoton	88	97	89	4	1
Ethyl methanesulfonate	79	109	97	1	3
Hexachloropropene	213	215	211	2	4
Isodrin	193	66	195	4	6
Isosafrole 1	162	104	131	3	2
Isosafrole 2	162	104	131	3	2
Methapyrilene	97	58	--	4	6
Methyl methanesulfonate	80	79	65	1	3
Methyl parathion	109	125	263	4	1
N-Nitrosodiethylamine	102	44	57	1	3
N-Nitrosodi-n-butylamine	84	57	41	2	4
N-Nitrosomethylethylamine	88	42	43	1	3
N-Nitrosomorpholine	116	56	86	1	3
N-Nitrosopiperidine	114	42	55	2	4
N-Nitrosopyrrolidine	100	41	42	1	3
O,O',O"-Triethyl-Phosphorothioate	198	121	93	2	4

Table 7. (continued)
Characteristic Ions, Appendix IX Standard (in alpha-numeric order)

Analyte	Primary	Secondary	Tertiary	IS	SURR
o-Toluidine	106	107	77	1	3
p-(Dimethylamino)azobenzene	120	225	77	5	6
Parathion	109	97	291	4	1
p-Chlorobenzilate	251	139	253	5	6
Pentachlorobenzene	250	248	252	3	2
Pentachloroethane	117	119	167	1	3
Pentachloronitrobenzene	237	142	214	4	1
Phenacetin	108	179	109	4	1
Phorate	75	97	121	4	1
p-Phenylenediamine	108	80	54	2	6
Pronamide	173	175	255	4	1
Safrole	162	104	77	2	4
Sulfotepp	97	322	202	4	1
Thionazin	97	96	143	3	2

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Table 8.
Characteristic Ions, Non-Routine Compounds[1]

Analyte	Primary	Secondary	Tertiary	IS	SURR	Curve ID
1,1'-Biphenyl	154	153	76	3	2	HSL
1,2-Dimethyl-3,4-Dinitrobenzene	179	91	77	4	1	DNX
1,2-Dimethyl-3,5-Dinitrobenzene	179	77	103	4	1	DNX
1,2-Dimethyl-3,6-Dinitrobenzene	179	77	133	3	1	DNX
1,2-Dimethyl-4,5-Dinitrobenzene	91	196	77	4	1	DNX
1,3-Dimethyl-2,4-Dinitrobenzene	77	179	91	3	1	DNX
1,3-Dimethyl-2,5-Dinitrobenzene	179	77	104	3	1	DNX
1,3-Phenylenediamine	108	80	81	1	2	PHE
1,4-Dimethyl-2,3-Dinitrobenzene	77	91	179	3	1	DNX
1,4-Dimethyl-2,6-Dinitrobenzene	179	77	91	3	1	DNX
1,5-Dimethyl-2,3-Dinitrobenzene	179	91	77	4	1	DNX
1,5-Dimethyl-2,4-Dinitrobenzene	179	77	91	3	1	DNX
1-Chloronaphthalene	162	127	164	3	2	AP9
1-Methylnaphthalene	142	141	115	2	4	HSL
2,3-Dichlorobenzenamine	161	163	90	3	2	HSL
2,4,6-Tribromophenol	329.9	331.9	141	3	1	HSL
2,4-Xylidine	121	120	106	2	4	PHE
2-Ethoxyethanol	59	45	72	1	3	CUST
3,5-Dimethylphenol	107	122	121	2	4	HSL
3-Methylphenol	108	107	77	1	3	HSL
4,4-Methylene bis(2-Chloroaniline)	231	266	238	5	6	AP9
5-Methyl-o-Anisidine	122	137	94	1	3	PHE
6-Methylchrysene	242	241	239	5	6	AP9
Acrylamide	71	54.9	44	1	3	AP9
Alachlor	188	160	45	1	1	HSL
Alpha Methyl Styrene	118	117	103	1	3	HSL
Alpha-Terpineol	59	93	121	1	4	REF
Atrazine	200	58	43	4	1	HSL
Benzaldehyde	106	105	77	1	5	BZHD
Benzo(e)pyrene	252	250	253	6	6	HSL
Benzyl dichloride	124.9	159.9	89	2	3	CUST
Caprolactam	55	113	84	2	4	HSL
Carbofuran Phenol	164	121.9	123	2	4	CUST
Dibenz(a,h)acridine	279	280	278	6	6	AP9
Dibenzo(a,e)pyrene	302	150	151	6	6	REF
Dimethylformamide	73	44	42	1	3	CUST

[1] Some non-routine analytes are not contained in the standard mixes. Appropriate calibrations are performed when these analytes are requested, along with assignment of IS and surrogate compounds at time of analysis.

Table 8. (continued)
Characteristic Ions, Non-Routine Compounds[1]

Analyte	Primar	Secondary	Tertiary	IS	SURR	Curve ID
Diphenylamine	169	168	167	3	2	AP9
Famphur	218	93	125	5	6	HSL
Hexachlorophene	196	198	--	5	6	HSL
Hexadecane	57	43	71	3	2	HSL
Indene	116	115	89	1	3	HSL
Isosafrole	162	131	104	3	2	AP9
Alpha Methyl Styrene	118	117	103	1	5	HSL
n-Decane	43	57	71	1	2	HSL
o-Anisidine	80	108	123	1	3	PHE
Perylene	252	253	126	6	6	CUST
Phenylmercaptan	110	77	109	1	3	REF
Phthalic Acid	148	149	76	3	2	REF
Phthalic Anhydride	76	104	50	3	2	REF
Quinoline	129	102	128	2	4	AP9
Toluene Diamine (2,4- + 2,6 –Isomers)	122	121	94	3	2	CUST
Tributyl Phosphate	99	155	211	3	1	CUST
Triethyl Amine	85.9	57.9	101	1	3	CUST
Triethyl Phosphate	99	155	127	2	4	CUST
Tris (2,3-dibromopropyl)phosphate	119	121	219	6	6	CUST

[1] Some non-routine analytes are not contained in the standard mixes. Appropriate calibrations are performed when these analytes are requested, along with assignment of IS and surrogate compounds at time of analysis.

Table 9.
8270C Surrogate Compounds

Surrogate Compounds	Standard Analysis ng/µL in extract	LVI Analysis ng/µL in extract
Nitrobenzene-d_5	100	25
2-Fluorobiphenyl	100	25
Terphenyl-d_{14}	100	25
1,2-Dichlorobenzene-d_4[1]	100	25
Phenol-d_5	100	25
2-Fluorophenol	100	25
2,4,6-Tribromophenol	100	25
2-Chlorophenol-d_4[1]	100	25

[1] Included in standard mix, but not evaluated or reported.

Recovery limits for surrogates are generated from historical data and are maintained by the QA department and stored in the LIMS

Table 10.
Calibration Levels[1], Primary (HSL) Standard, µg/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	Level 8
Pyridine	-	10	20	50	80	120	160	200
1,4-Dioxane	4	10	20	50	80	120	160	200
N-nitrosodimethylamine	-	10	20	50	80	120	160	200
Aniline	-	10	20	50	80	120	160	200
Phenol	-	10	20	50	80	120	160	200
Bis(2-chloroethyl)ether	-	10	20	50	80	120	160	200
2-Chlorophenol	-	10	20	50	80	120	160	200
1,3-Dichlorobenzene	4	10	20	50	80	120	160	200
1,4-Dichlorobenzene	4	10	20	50	80	120	160	200
Benzyl alcohol	-	10	20	50	80	120	160	200
1,2-Dichlorobenzene	4	10	20	50	80	120	160	200
2-Methylphenol	-	10	20	50	80	120	160	200
2,2'-oxybis(1-chloropropane)[2]	-	10	20	50	80	120	160	200
4-Methylphenol	-	10	20	50	80	120	160	200
3-Methylphenol	-	10	20	50	80	120	160	200
Acetophenone	4	10	20	50	80	120	160	200
N-Nitroso-di-n-propylamine	-	10	20	50	80	120	160	200
Hexachloroethane	4	10	20	50	80	120	160	200
Nitrobenzene	-	10	20	50	80	120	160	200
Isophorone	-	10	20	50	80	120	160	200
2-Nitrophenol	-	10	20	50	80	120	160	200
2,4-Dimethylphenol	-	10	20	50	80	120	160	200
Benzoic acid	-	--	20	50	80	120	160	200
Bis(2-chloroethoxy)methane	-	10	20	50	80	120	160	200
2,4-Dichlorophenol	-	10	20	50	80	120	160	200
1,2,4-Trichlorobenzene	4	10	20	50	80	120	160	200
Naphthalene	4	10	20	50	80	120	160	200
4-Chloroaniline	-	10	20	50	80	120	160	200
Hexachlorobutadiene	-	10	20	50	80	120	160	200
1,2,4,5-Tetrachlorobenzene	-	10	20	50	80	120	160	200
4-Chloro-3-methylphenol	-	10	20	50	80	120	160	200
2-Methylnaphthalene	4	10	20	50	80	120	160	200
Hexachlorocyclopentadiene	-	--	20	50	80	120	160	200
2,4,6-Trichlorophenol	-	10	20	50	80	120	160	200
2,4,5-Trichlorophenol	-	10	20	50	80	120	160	200
2-Chloronaphthalene	4	10	20	50	80	120	160	200
2-Nitroaniline	-	--	20	50	80	120	160	200
Dimethyl phthalate	4	10	20	50	80	120	160	200
1,3-Dinitrobenzene	-	10	20	50	80	120	160	200
Acenaphthylene	4	10	20	50	80	120	160	200
3-Nitroaniline	-	--	20	50	80	120	160	200
Acenaphthene	4	10	20	50	80	120	160	200
2,4-Dinitrophenol	-	20	40	100	160	240	320	400
4-Nitrophenol	8	20	40	100	160	240	320	400
Dibenzofuran	4	10	20	50	80	120	160	200
2,3,4,6-Tetrachlorophenol	-	10	20	50	80	120	160	200
2,4-Dinitrotoluene	-	10	20	50	80	120	160	200
2,6-Dinitrotoluene	-	10	20	50	80	120	160	200

Table 10. (continued)
Calibration Levels, Primary (HSL) Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Diethylphthalate	4	10	20	50	80	120	160	200
4-Chlorophenyl phenyl ether	-	10	20	50	80	120	160	200
Fluorene	4	10	20	50	80	120	160	200
4-Nitroaniline	-	--	20	50	80	120	160	200
4,6-Dinitro-2-methylphenol	-	20	40	100	160	240	320	400
N-Nitrosodiphenylamine	-	10	20	50	80	120	160	200
Azobenzene[3]	4	10	20	50	80	120	160	200
4-Bromophenyl phenyl ether	-	10	20	50	80	120	160	200
Hexachlorobenzene	-	10	20	50	80	120	160	200
Pentachlorophenol	-	20	40	100	160	240	320	400
Phenanthrene	4	10	20	50	80	120	160	200
Anthracene	4	10	20	50	80	120	160	200
Carbazole	4	10	20	50	80	120	160	200
Di-n-butyl phthalate	4	10	20	50	80	120	160	200
Fluoranthene	4	10	20	50	80	120	160	200
Benzidine	-	--	20	50	80	120	160	200
Pyrene	4	10	20	50	80	120	160	200
Butyl benzyl phthalate	4	10	20	50	80	120	160	200
3,3'-Dichlorobenzidine	-	--	20	50	80	120	160	200
Benzo(a)anthracene	4	10	20	50	80	120	160	200
Bis(2-ethylhexyl)phthalate	4	10	20	50	80	120	160	200
Chrysene	4	10	20	50	80	120	160	200
Di-n-octylphthalate	4	10	20	50	80	120	160	200
Benzo(b)fluoranthene	4	10	20	50	80	120	160	200
Benzo(k)fluoranthene	4	10	20	50	80	120	160	200
Benzo(a)pyrene	4	10	20	50	80	120	160	200
Indeno(1,2,3-cd)pyrene	4	10	20	50	80	120	160	200
Dibenz(a,h)anthracene	4	10	20	50	80	120	160	200
Benzo(g,h,i)perylene	4	10	20	50	80	120	160	200

1. All compounds are spiked in all standard levels (4, 10, 20, 50, 80, 120, 160 and 200 µg/mL). Levels listed in this table are the required spike levels needed in order to calibrate to at the reporting limit for each compound. Additional lower-level standards may be included in the calibrations as long as the ICAL criteria are met.
2. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
3. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table 11.
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	10	20	50	80	120	160	200
N-Nitrosomethylethylamine	10	20	50	80	120	160	200
Methyl methanesulfonate	10	20	50	80	120	160	200
N-Nitrosodiethylamine	10	20	50	80	120	160	200
Ethyl methanesulfonate	10	20	50	80	120	160	200
Pentachloroethane	--	20	50	80	120	160	200
N-Nitrosopyrrolidine	10	20	50	80	120	160	200
N-Nitrosomorpholine	10	20	50	80	120	160	200
o-Toluidine	10	20	50	80	120	160	200
N-Nitrosopiperidine	10	20	50	80	120	160	200
O,O',O"-Triethyl-Phosphorothioate	--	20	50	80	120	160	200
a,a-Dimethyl-phenethylamine	--	20	50	80	120	160	200
2,6-Dichlorophenol	10	20	50	80	120	160	200
Hexachloropropene	--	20	50	80	120	160	200
p-Phenylenediamine	--	20	50	80	120	160	200
n-Nitrosodi-n-butylamine	10	20	50	80	120	160	200
Safrole	--	20	50	80	120	160	200
Isosafrole 1 + 2	10	20	50	80	120	160	200
1,4-Dinitrobenzene	10	20	50	80	120	160	200
1,4-Naphthoquinone	--	20	50	80	120	160	200
Pentachlorobenzene	10	20	50	80	120	160	200
1-Naphthylamine	10	20	50	80	120	160	200
2-Naphthylamine	10	20	50	80	120	160	200
N-Nitro-o-toluidine	10	20	50	80	120	160	200
Thionazin	10	20	50	80	120	160	200
1,3,5-Trinitrobenzene	--	20	50	80	120	160	200
Sulfotepp	--	20	50	80	120	160	200
Phorate	--	20	50	80	120	160	200
Phenacetin	10	20	50	80	120	160	200
Diallate 1 + 2	10	20	50	80	120	160	200
Dimethoate	10	20	50	80	120	160	200
4-Aminobiphenyl	--	20	50	80	120	160	200
Pentachloronitrobenzene	--	20	50	80	120	160	200
Pronamide	10	20	50	80	120	160	200
Disulfoton	--	20	50	80	120	160	200
2-secbutyl-4,6-dinitrophenol (Dinoseb)	10	20	50	80	120	160	200
Methyl parathion	--	20	50	80	120	160	200
4-Nitroquinoline-1-oxide	--	20	50	80	120	160	200
Parathion	--	20	50	80	120	160	200

Table 11. (continued)
Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	10	20	50	80	120	160	200
Methapyrilene	--	20	50	80	120	160	200
Aramite 1 and 2	10	20	50	80	120	160	200
p-(Dimethylamino) azobenzene	10	20	50	80	120	160	200
p-Chlorobenzilate	10	20	50	80	120	160	200
3,3'-Dimethylbenzidine	10	20	50	80	120	160	200
2-Acetylaminofluorene	--	20	50	80	120	160	200
Dibenz (a,j)acridine	10	20	50	80	120	160	200
7,12-Dimethylbenz(a) anthracene	10	20	50	80	120	160	200
3-Methylcholanthrene	10	20	50	80	120	160	200

Table 12.
Calibration Levels, Non-routine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
1,1'-Biphenyl	--	10	20	50	80	120	160	200	HSL
1,2-Dimethyl-3,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-3,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-3,6-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,2-Dimethyl-4,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Dimethyl-2,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Dimethyl-2,5-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,3-Phenylenediamine	--	10	20	50	80	120	160	200	PHE
1,4-Dimethyl-2,3-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,4-Dimethyl-2,6-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,5-Dimethyl-2,3-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1,5-Dimethyl-2,4-Dinitrobenzene	4	10	20	50	80	120	160	200	DNX
1-Chloronaphthalene	--	10	20	50	80	120	160	200	AP9
1-Methylnaphthalene	4	10	20	50	80	120	160	200	HSL
2,3-Dichlorobenzenamine	4	10	20	50	80	120	160	200	HSL
2,4,6-Tribromophenol	4	10	20	50	80	120	160	200	HSL
2,4-Xylidine	--	10	20	50	80	120	160	200	PHE
2-Ethoxyethanol	--	10	20	50	80	120	160	200	CUST
3,5-Dimethylphenol	4	10	20	50	80	120	160	200	HSL
3-Methylphenol	4	10	20	50	80	120	160	200	HSL
4,4-Dichlorobenzil	--	10	20	50	80	120	160	200	BRC
4,4-Methylene bis(2-Chloroaniline)	--	10	20	50	80	120	160	200	AP9
4-Chlorobenzenethiol	--	10	20	50	80	120	160	200	BRC
5-Methyl-o-Anisidine	--	10	20	50	80	120	160	200	PHE

Table 12. (continued)
Calibration Levels, Non-routine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
6-Methylchrysene	--	10	20	50	80	120	160	200	AP9
Acrylamide	--	10	20	50	80	120	160	200	AP9
Alachlor	4	10	20	50	80	120	160	200	HSL
Alpha Methyl Styrene	4	10	20	50	80	120	160	200	HSL
Alpha-Terpineol	--	10	20	50	80	120	160	200	REF
Atrazine	4	10	20	50	80	120	160	200	HSL
Benzaldehyde	--	10	20	50	80	120	160	200	BZHD
Benzo(e)pyrene	4	10	20	50	80	120	160	200	HSL
Benzyl dichloride	--	10	20	50	80	120	160	200	CUST
Bis(4-Chlorophenyl)disulfide	--	10	20	50	80	120	160	200	BRC
Caprolactam	4	10	20	50	80	120	160	200	HSL
Carbofuran Phenol	--	10	20	50	80	120	160	200	CUST
Dibenz(a,h)acridine	--	10	20	50	80	120	160	200	AP9
Dibenzo(a,e)pyrene	--	10	20	50	80	120	160	200	REF
Dimethylformamide	--	10	20	50	80	120	160	200	CUST
Diphenylamine	--	10	20	50	80	120	160	200	AP9
Diphenylsulfone	--	10	20	50	80	120	160	200	BRC
Famphur	4	10	20	50	80	120	160	200	HSL
Hexachlorophene	4	10	20	50	80	120	160	200	HSL
Hexadecane	4	10	20	50	80	120	160	200	HSL
Indene	4	10	20	50	80	120	160	200	HSL
Isosafrole	--	10	20	50	80	120	160	200	AP9
Methyl 4-Chlorophenyl Sulfide	--	10	20	50	80	120	160	200	BRC
n-Decane	4	10	20	50	80	120	160	200	HSL
n-Hydroxymethlphthalimide	4	10	20	50	80	120	160	200	HSL
o-Anisidine	--	10	20	50	80	120	160	200	PHE
Octachlorostyrene	--	10	20	50	80	120	160	200	BRC
p-Chlorophenyl Sulfone	--	10	20	50	80	120	160	200	BRC
Perylene	--	10	20	50	80	120	160	200	CUST
Phenyl Disulfide	--	10	20	50	80	120	160	200	BRC
Phenyl Sulfide	--	10	20	50	80	120	160	200	BRC
Phenylmercaptan	--	10	20	50	80	120	160	200	REF
Phthalic Acid	--	10	20	50	80	120	160	200	REF
Phthalic Anhydride	--	10	20	50	80	120	160	200	REF
Quinoline	--	10	20	50	80	120	160	200	AP9
Toluene Diamine (2,4- + 2,6 –	--	10	20	50	80	120	160	200	CUST
Tributyl Phosphate	--	10	20	50	80	120	160	200	CUST
Triethyl Amine	--	10	20	50	80	120	160	200	CUST
Triethyl Phosphate	--	10	20	50	80	120	160	200	CUST
Tris (2,3-dibromopropyl)phosphate	--	10	20	50	80	120	160	200	CUST

COMPANY CONFIDENTIAL AND PROPRIETARY

Table 13.
List 1 – Reliably Performing Compounds

Acenaphthene	Dibenzofuran	1H-Indene
Acenaphthylene	1,4-Dioxane	Indeno(1,2,3-cd)pyrene
Acetophenone	n-Dodecane	Isophorone
Alachlor	n-Docosane	1-Methylnaphthalene
Aniline	1,2-Dichlorobenzene	2-Methylnaphthalene
Anthracene	1,3-Dichlorobenzene	2-Methylphenol
Atrazine	1,4-Dichlorobenzene	4-Methylphenol
Benzo(a)anthracene	2,3-Dichlorobenzeneamine	Methylstyrene
Benzo(a)pyrene	3,3'-Dichlorobenzidine	Naphthalene
Benzo(b)fluoranthene	2,4-Dichlorophenol	2-Nitroaniline
Benzo(k)fluoranthene	Diethyl phthalate	3-Nitroaniline
Benzo(g,h,i)perylene	2,4-Dimethylphenol	4-Nitroaniline
Benzoic acid	Dimethyl phthalate	Nitrobenzene
Benzyl alcohol	Di-n-butyl phthalate	2-Nitrophenol
Bis(2-chloroethoxy)methane	4,6-Dinitro-2-methylphenol	4-Nitrophenol
Bis(2-chloroethyl)ether	2,4-Dinitrophenol	N-Nitrosodimethylamine
Bis(2-ethylhexyl)phthalate	2,4-Dinitrotoluene	N-Nitroso-di-n-propylamine
4-Bromophenyl phenyl ether	2,6-Dinitrotoluene	N-Nitrosodiphenylamine
Butyl benzyl phthalate	1,2-Diphenylhydrazine (as Azobenzene)	2,2'-Oxybis(1-chloropropane) aka "bis(2-chloroisopropyl) ether"
Caprolactam	Di-n-octyl phthalate	n-Octadecane
Carbazole	n-Eicosane	Pentachlorophenol
4-Chloroaniline	Famphur	Phenanthrene
4-Chloro-3-methylphenol	Fluoranthene	Phenol
2-Chloronaphthalene	Fluorene	Pyrene
2-Chlorophenol	Hexachlorobenzene	Pyridine
4-Chlorophenyl phenyl ether	Hexachlorocyclopentadiene	n-Tetradecane
Chrysene	Hexachlorobutadiene	1,2,4-Trichlorobenzene
n-Decane	Hexachloroethane	2,4,5-Trichlorophenol
Dibenz(a,h)anthracene	n-Hexadecane	2,4,6-Trichlorophenol

Table 14.
List 2 – Poorly Performing Compounds

2-Acetylaminofluorene	Diphenylamine	N-Nitrosopyrrolidine
Acrylamide	Disulfoton	Parathion
4-Aminobiphenyl	2-Ethoxyethanol	Pentachlorobenzene
Aramite (#1)	Ethyl methanesulfonate	Pentachloroethane
Aramite (#2)	Hexachlorophene	Pentachloronitrobenzene
Benzenethiol	Hexachloropropene	Perylene
Benzidine	Isosafrole (#1)	Phenacetin
Benzyl chloride	Isosafrole (#2)	p-Phenylenediamine
Biphenyl	Isodrin	Phorate
Carbofuran phenol	Methapyrilene	Phthalic anhydride
Chlorobenzilate	Methomyl	2-Picoline
Diallate (#1)	3-Methylcholanthrene	Pronamide
Diallate (#2)	6-Methylchrysene	Quinoline
Dibenz(a,h)acridine	4,4"-Methylenebis(2-chloroaniline)	Safrole
Dibenz(a,j)acridine	Methyl methanesulfonate	2-secbutyl-4,6-dinitrophenol (Dinoseb)
Dibenzo(a,e)pyrene	Methyl Parathion	Sulfotepp
Tris(2,3-Dibromopropyl) phosphate	1-Naphthylamine	1,2,4,5-Tetrachlorobenzene
2,6-Dichlorophenol	2-Naphthylamine	2,3,4,6-Tetrachlorophenol
Dimethoate	1,4-Naphthoquinone	Thionazin
p-(Dimethylamino)azobenzene	5-Nitro-o-toluidine	o-Toluidine
7,12-Dimethylbenz(a)anthracene	4-Nitroquinoline-1-oxide	2,4- and 2,6-Toluenediamine
3,3'-Dimethylbenzidine	N-Nitrosodiethylamine	Triethylamine
N,N-Dimtheylformamide	n-Nitrosodi-n-butylamine	Triethylphosphate
a,a-Dimethyl-phenethylamine	N-Nitrosomethylethylamine	o,o,o-Triethylphosphorothioate
1,3-Dinitrobenzene	N-Nitrosomorpholine	1,3,5-Trinitrobenzene
1,4-Dinitrobenzene	N-Nitrosopiperidine	

Table 15
DoD QSM 5.0 GCMS Method Requirements

Requirement	*QSM 5.0 Table 4. Organic Analysis by Gas Chromatography/Mass Spectrometry*
Tune Check	Perform tune check prior to calibration and prior to each period of every 12 hours during sample analysis. Use method specified ion abundance criteria for BFB or DFTPP. If unsuccessful, retune and verify. No samples shall be analyzed without a valid tune.
Performance Check (Method 8270 only)	Performed daily at the beginning of each 12-hour period prior to analysis of samples. Degradation must be ≤20% for DDT. Any problems must be corrected. Do not run samples until degradation is ≤ 20%. Benzidine and pentachlorophenol should be present at their normal responses and peak tailing should not exceed a tailing factor of 2. If criteria not met correct problem and reanalyze.
Initial Calibration (ICAL) for all analytes including surrogates	Perform a minimum 5 point calibration for all analytes prior to sample analysis. (Minimum 6 points for non-linear). Acceptance Criteria options: Option 1: RSD for each analyte ≤ 15% Option 2: Linear least squares regression for each analyte: $r^2 \geq 0.99$ ($r \geq 0.995$) Option 3: Non-linear least square regression (quadratic) for each analyte, $r^2 \geq 0.99$. Additional method requirements for evaluation must be met, e.g., CCCs, SPCCs, low calibration standard analysis and recovery criteria, etc.) Any problems must be corrected and ICAL repeated. No sample can be run until ICAL is successful.
Retention Time (RT) window position establishment	Establish the RT window position for each analyte and surrogate once per ICAL and at the beginning of the analytical sequence. Set position using the midpoint standard of the calibration curve when an ICAL is performed. On days when ICAL is not performed the initial CCV is used.
Relative Retention Time (RRT) Verification	Evaluate RRTs for each sample compared with the most recently updated RRTs, based on the daily CCV. RRT of each target analyte in each calibration standard must be within ± 0.06 RRT units. Correct any problems, then rerun ICAL.
Initial Calibration Verification (ICV)	Measure a second-source standard once after each ICAL, prior to sample analysis. All analytes must be within ± 20% of the true value. Any problems must be corrected. No samples shall be analyzed until the second-source calibration verification is successful. *When allowed by the client the following technical specification may be used:* Allow ± 30% of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client for the project under consideration. (4PP)
Continuing calibration Verification (CCV)	CCV concentration ≤ midpoint of calibration range. Run before sample analysis and every 12 hours of analysis time and at the end of the analytical batch (end of 12 hours). Acceptance limits all analytes ± 20% of true value for CCV at start of 12 hours. Additional evaluation that must be met for the CCV to be acceptable, e.g., criteria for CCC and

Requirement	*QSM 5.0 Table 4. Organic Analysis by Gas Chromatography/Mass Spectrometry*
	SPCC compounds. Closing CCV: All reported analytes and surrogates within ± 20%. If any analytes fail in a CCV, recalibrate and re-analyze all affected samples or immediately (within one hour) analyze two consecutive CCVs and if both pass for the analytes that failed, the CCV is acceptable. Sample analysis may continue or previously analyzed samples may be reported without reanalysis. If acceptable CCV results cannot be obtained, repeat the ICAL. Apply Q-flag if no sample material remains and analyte fails criteria. *The following technical specification may be applied if approved by the client:* Allow ±30% of true value for known poor performers only if these compounds are not identified as critical compounds of concern by the client for the project under consideration. (4PP) If the CCV is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Allow ± 50% for end of analytical batch excluding poor performing compounds. Reanalysis performed due to failed closing CCV only for the analytes identified by the client as critical compounds of concern for the project, and to report qualified results for other analytes. (7MS)
Internal Standards (IS)	Use internal standard in all field samples, QC samples and standards. RT must be ± 10 seconds from RT of the midpoint standard in the ICAL. EICP area must be within -50% to 100% of ICAL midpoint standard. For any failures, inspect mass spec and GC for malfunctions. Samples analyzed while measurement system was malfunctioning must be reanalyzed. If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging is not appropriate for failed standards. *The following technical specification may be applied if approved by the client:* RT must be ± 30 seconds from RT of the midpoint standard in the ICAL. Daily routine column maintenance often results in larger RT changes than 10 seconds within a short time (8ISRT).
Method Blank (MB)	One per prep batch. No analytes detected > ½ LOQ (RL) or >1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. No common lab contaminants >LOQ The laboratory has the following GC/MS Semi-volatiles (8270) phthalate esters noted as common lab contaminants: bis-2-ethylhexylphthalate, di-n-octyl phthalate, butyl benzyl phthalate, di-n-butyl phthalate If criteria not met, correct problem. If required, reprep and reanalyze MB and all samples processed with the contaminated blank. If reanalysis is not possible, apply B-flag to all results for the specific analyte(s) in all samples processed with the contaminated blank. Must be explained in the case narrative. Flagging is only appropriate when samples cannot be reanalyzed.
LCS	One per prep batch. Include all analyte(s) in LCS that are required to be reported,

Requirement	*QSM 5.0 Table 4. Organic Analysis by Gas Chromatography/Mass Spectrometry*
	including surrogates. Use DoD acceptance criteria if project limits are not specified. If analyte not listed by DoD, use in-house LCS limits if project limits not specified. Marginal exceedances are not allowed for critical chemicals of concern (risk drivers). Client must notify TestAmerica of these targets or if marginal exceedances will not be allowed. Otherwise, correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Flagging is only appropriate when samples cannot be reanalyzed. *The following technical specification may be applied if approved by the client:* If the LCS recovery is above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project (3HR). Otherwise, correct any problems then re-prep and reanalyze the LCS and all associated samples for failed analytes. If insufficient sample, then apply Q-flag to specific analyte(s) in all samples in the associated prep batch. Flagging is only appropriate when samples cannot be reanalyzed unless 3HR is accepted by the client. One per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates, except those compounds listed as "Additional Analytes" by TestAmerica. These compounds are rarely requested and historical limits may not accurately reflect current performance. (4PP)
Matrix Spike	One per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates. Use DoD-specific criteria for LCS. If DoD limits are not available, use in-house LCS limits if project limits are not specified. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error. *The following technical specification may be applied if approved by the client:* Include all analyte(s) that are required to be reported, including surrogates, except those compounds listed as "Additional Analytes" by TestAmerica. These compounds are rarely requested and historical limits may not accurately reflect current performance. (4PP)
MSD or Sample Duplicate	One per prep batch. Include all analyte(s) in LCS that are required to be reported, including surrogates. Use DoD-specific criteria for LCS. If DoD limits are not available, use in-house LCS limits if project limits are not specified. For failures, consult project-specific DQOs and contact client for additional measures to be taken. For specific analyte(s) in parent sample, apply J-flag if acceptance criteria are not met. If MS falls outside LCS limits, evaluate data to determine the source of the difference and to determine if there is a matrix effect or analytical error. MSD or MD: RPD of all analytes $\leq$ 20% (between MS and MSD or sample and MD) *The following technical specification may be applied if approved by the client:* Include all analyte(s) that are required to be reported, including surrogates, except those compounds listed as "Additional Analytes" by TestAmerica. These compounds are rarely requested and historical limits may not accurately reflect current performance. (4PP)

Requirement	*QSM 5.0 Table 4. Organic Analysis by Gas Chromatography/Mass Spectrometry*
Surrogates	Spike all field and QC samples with surrogates. Must meet DoD acceptance criteria, otherwise method-specified criteria or lab's in-house criteria. For QC and field samples, correct any problems, then re-prep and reanalyze all failed samples for failed surrogates in the associated prep batch. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the case narrative. All surrogates analyzed must be reported. *The following technical specification may be applied if approved by the client:* If surrogate recoveries are above the project acceptance limits and there are no detections in the samples, TestAmerica will report the non-detect results with a case narrative comment in addition to applying any data qualifier flags required by the project. (3HR).

Table LVI-1.
LVI Calibration Levels, Primary (HSL) Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Pyridine	1	2.5	5	12.5	20	30	40	50
N-nitrosodimethylamine	1	2.5	5	12.5	20	30	40	50
Aniline	1	2.5	5	12.5	20	30	40	50
Phenol	1	2.5	5	12.5	20	30	40	50
Bis(2-chloroethyl)ether	1	2.5	5	12.5	20	30	40	50
2-Chlorophenol	1	2.5	5	12.5	20	30	40	50
1,3-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
1,4-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
Benzyl alcohol	1	2.5	5	12.5	20	30	40	50
1,2-Dichlorobenzene	1	2.5	5	12.5	20	30	40	50
2-Methylphenol	1	2.5	5	12.5	20	30	40	50
2,2'-oxybis(1-chloropropane)[1]	1	2.5	5	12.5	20	30	40	50
4-Methylphenol	1	2.5	5	12.5	20	30	40	50
3-Methylphenol	1	2.5	5	12.5	20	30	40	50
Acetophenone	1	2.5	5	12.5	20	30	40	50
N-Nitroso-di-n-propylamine	1	2.5	5	12.5	20	30	40	50
Hexachloroethane	1	2.5	5	12.5	20	30	40	50
Nitrobenzene	1	2.5	5	12.5	20	30	40	50
Isophorone	1	2.5	5	12.5	20	30	40	50
2-Nitrophenol	1	2.5	5	12.5	20	30	40	50
2,4-Dimethylphenol	1	2.5	5	12.5	20	30	40	50
Benzoic acid	2	5	10	25	40	60	80	100
Bis(2-chloroethoxy)methane	1	2.5	5	12.5	20	30	40	50
2,4-Dichlorophenol	1	2.5	5	12.5	20	30	40	50
1,2,4-Trichlorobenzene	1	2.5	5	12.5	20	30	40	50
Naphthalene	1	2.5	5	12.5	20	30	40	50
4-Chloroaniline	1	2.5	5	12.5	20	30	40	50
Hexachlorobutadiene	1	2.5	5	12.5	20	30	40	50
4-Chloro-3-methylphenol	1	2.5	5	12.5	20	30	40	50
2-Methylnaphthalene	1	2.5	5	12.5	20	30	40	50
Hexachlorocyclopentadiene	1	2.5	5	12.5	20	30	40	50
1,2,4,5-Tetrachlorobenzene	1	2.5	5	12.5	20	30	40	50
2,4,6-Trichlorophenol	1	2.5	5	12.5	20	30	40	50
2,4,5-Trichlorophenol	1	2.5	5	12.5	20	30	40	50
2-Chloronaphthalene	1	2.5	5	12.5	20	30	40	50
2-Nitroaniline	1	2.5	5	12.5	20	30	40	50
Dimethyl phthalate	1	2.5	5	12.5	20	30	40	50
1,3-Dinitrobenzene	1	2.5	5	12.5	20	30	40	50
Acenaphthylene	1	2.5	5	12.5	20	30	40	50
3-Nitroaniline	1	2.5	5	12.5	20	30	40	50
Acenaphthene	1	2.5	5	12.5	20	30	40	50
2,4-Dinitrophenol	2	5	10	25	40	60	80	100
4-Nitrophenol	2	5	10	25	40	60	80	100
Dibenzofuran	1	2.5	5	12.5	20	30	40	50
2,3,4,6-Tetrachlorophenol	1	2.5	5	12.5	20	30	40	50
2,4-Dinitrotoluene	1	2.5	5	12.5	20	30	40	50
2,6-Dinitrotoluene	1	2.5	5	12.5	20	30	40	50

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Table LVI-1. (continued)
LVI Calibration Levels, Primary Standard, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8
Diethylphthalate	1	2.5	5	12.5	20	30	40	50
4-Chlorophenyl phenyl ether	1	2.5	5	12.5	20	30	40	50
Fluorene	1	2.5	5	12.5	20	30	40	50
4-Nitroaniline	1	2.5	5	12.5	20	30	40	50
4,6-Dinitro-2-methylphenol	2	5	10	25	40	60	80	100
N-Nitrosodiphenylamine	1	2.5	5	12.5	20	30	40	50
Azobenzene[2]	1	2.5	5	12.5	20	30	40	50
4-Bromophenyl phenyl ether	1	2.5	5	12.5	20	30	40	50
Hexachlorobenzene	1	2.5	5	12.5	20	30	40	50
Pentachlorophenol	2	5	10	25	40	60	80	100
Phenanthrene	1	2.5	5	12.5	20	30	40	50
Anthracene	1	2.5	5	12.5	20	30	40	50
Carbazole	1	2.5	5	12.5	20	30	40	50
Di-n-butyl phthalate	1	2.5	5	12.5	20	30	40	50
Fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzidine	1	2.5	5	12.5	20	30	40	50
Pyrene	1	2.5	5	12.5	20	30	40	50
Butyl benzyl phthalate	1	2.5	5	12.5	20	30	40	50
3,3'-Dichlorobenzidine	1	2.5	5	12.5	20	30	40	50
Benzo(a)anthracene	1	2.5	5	12.5	20	30	40	50
Bis(2-ethylhexyl)phthalate	1	2.5	5	12.5	20	30	40	50
Chrysene	1	2.5	5	12.5	20	30	40	50
Di-n-octylphthalate	1	2.5	5	12.5	20	30	40	50
Benzo(b)fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzo(k)fluoranthene	1	2.5	5	12.5	20	30	40	50
Benzo(a)pyrene	1	2.5	5	12.5	20	30	40	50
Indeno(1,2,3-cd)pyrene	1	2.5	5	12.5	20	30	40	50
Dibenz(a,h)anthracene	1	2.5	5	12.5	20	30	40	50
Benzo(g,h,i)perylene	1	2.5	5	12.5	20	30	40	50

1. 2,2'oxybis(1-chloropropane) was formally known as bis(2-chloroisopropyl)ether
2. Azobenzene is formed by decomposition of 1,2-diphenlyhydrazine. If 1,2-diphenylhydrazine is requested, it will be analyzed as azobenzene.

Table LVI-2.
LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
2-Picoline	2.5	5	12.5	20	30	40	50
N-Nitrosomethylethylamine	2.5	5	12.5	20	30	40	50
Methyl methanesulfonate	2.5	5	12.5	20	30	40	50
N-Nitrosodiethylamine	2.5	5	12.5	20	30	40	50
Ethyl methanesulfonate	2.5	5	12.5	20	30	40	50
Pentachloroethane	2.5	5	12.5	20	30	40	50
N-Nitrosopyrrolidine	2.5	5	12.5	20	30	40	50
N-Nitrosomorpholine	2.5	5	12.5	20	30	40	50
o-Toluidine	2.5	5	12.5	20	30	40	50
N-Nitrosopiperidine	2.5	5	12.5	20	30	40	50
O,O',O"-Triethyl-Phosphorothioate	2.5	5	12.5	20	30	40	50
a,a-Dimethyl-phenethylamine	2.5	5	12.5	20	30	40	50
2,6-Dichlorophenol	2.5	5	12.5	20	30	40	50
Hexachloropropene	2.5	5	12.5	20	30	40	50
p-Phenylenediamine	2.5	5	12.5	20	30	40	50
n-Nitrosodi-n-butylamine	2.5	5	12.5	20	30	40	50
Safrole	2.5	5	12.5	20	30	40	50
Isosafrole 1 + 2	2.5	5	12.5	20	30	40	50
1,4-Dinitrobenzene	2.5	5	12.5	20	30	40	50
1,4-Naphthoquinone	2.5	5	12.5	20	30	40	50
Pentachlorobenzene	2.5	5	12.5	20	30	40	50
1-Naphthylamine	2.5	5	12.5	20	30	40	50
2-Naphthylamine	2.5	5	12.5	20	30	40	50
N-Nitro-o-toluidine	2.5	5	12.5	20	30	40	50
Thionazin	2.5	5	12.5	20	30	40	50
1,3,5-Trinitrobenzene	2.5	5	12.5	20	30	40	50
Sulfotepp	2.5	5	12.5	20	30	40	50
Phorate	2.5	5	12.5	20	30	40	50
Phenacetin	2.5	5	12.5	20	30	40	50
Diallate 1 + 2	2.5	5	12.5	20	30	40	50
Dimethoate	2.5	5	12.5	20	30	40	50
4-Aminobiphenyl	2.5	5	12.5	20	30	40	50
Pentachloronitrobenzene	2.5	5	12.5	20	30	40	50
Pronamide	2.5	5	12.5	20	30	40	50
Disulfoton	2.5	5	12.5	20	30	40	50
2-secbutyl-4,6-dinitrophenol (Dinoseb)	2.5	5	12.5	20	30	40	50
Methyl parathion	2.5	5	12.5	20	30	40	50
4-Nitroquinoline-1-oxide	2.5	5	12.5	20	30	40	50
Parathion	2.5	5	12.5	20	30	40	50

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Table LVI-2. (continued)
LVI Calibration Levels, Appendix IX Standard, µg/mL

Semivolatiles	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7
Isodrin	2.5	5	12.5	20	30	40	50
Methapyrilene	2.5	5	12.5	20	30	40	50
Aramite 1 and 2	2.5	5	12.5	20	30	40	50
p-(Dimethylamino) azobenzene	2.5	5	12.5	20	30	40	50
p-Chlorobenzilate	2.5	5	12.5	20	30	40	50
3,3'-Dimethylbenzidine	2.5	5	12.5	20	30	40	50
2-Acetylaminofluorene	2.5	5	12.5	20	30	40	50
Dibenz (a,j)acridine	2.5	5	12.5	20	30	40	50
7,12-Dimethylbenz(a) anthracene	2.5	5	12.5	20	30	40	50
3-Methylcholanthrene	2.5	5	12.5	20	30	40	50

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Table LVI-3.
LVI Calibration Levels, Non-routine compounds, µg/mL

Analyte	L1	L2	L3	L4	L5	L6	L7	L8	CURVE ID
1,1'-Biphenyl	--	2.5	5	12.5	20	30	40	50	HSL
1-Chloronaphthalene	--	2.5	5	12.5	20	30	40	50	AP9
1-Methylnaphthalene	1	2.5	5	12.5	20	30	40	50	HSL
2,3-Dichlorobenzenamine	1	2.5	5	12.5	20	30	40	50	HSL
2,4,6-Tribromophenol	1	2.5	5	12.5	20	30	40	50	HSL
3,5-Dimethylphenol	1	2.5	5	12.5	20	30	40	50	HSL
3-Methylphenol	1	2.5	5	12.5	20	30	40	50	HSL
4,4-Dichlorobenzil	1	2.5	5	12.5	20	30	40	50	BRC
4,4'-Methylene bis(2-chloroaniline)	1	2.5	5	12.5	20	30	40	50	AP9
4-Chlorobenzenethiol	1	2.5	5	12.5	20	30	40	50	BRC
6-Methylchrysene	1	2.5	5	12.5	20	30	40	50	AP9
Acrylamide	1	2.5	5	12.5	20	30	40	50	AP9
Alachlor	1	2.5	5	12.5	20	30	40	50	HSL
Alpha Methyl Styrene	1	2.5	5	12.5	20	30	40	50	HSL
Alpha-Terpineol	1	2.5	5	12.5	20	30	40	50	REF
Atrazine	1	2.5	5	12.5	20	30	40	50	HSL
Benzaldehyde	1	2.5	5	12.5	20	30	40	50	BZHD
Benzo(e)pyrene	1	2.5	5	12.5	20	30	40	50	HSL
Bis(4-Chlorophenyl)disulfide	1	2.5	5	12.5	20	30	40	50	BRC
Caprolactam	1	2.5	5	12.5	20	30	40	50	HSL
Dibenz(a,h)acridine	1	2.5	5	12.5	20	30	40	50	AP9
Dibenz(a,e)pyrene	1	2.5	5	12.5	20	30	40	50	REF
Diphenylamine	--	2.5	5	12.5	20	30	40	50	AP9
Diphenylsulfone	1	2.5	5	12.5	20	30	40	50	BRC
Famphur	1	2.5	5	12.5	20	30	40	50	HSL
Hexachlorophene	1	2.5	5	12.5	20	30	40	50	HSL
Hexadecane	1	2.5	5	12.5	20	30	40	50	HSL
Indene	1	2.5	5	12.5	20	30	40	50	HSL
Isosafrole	--	2.5	5	12.5	20	30	40	50	AP9
Methyl 4-Chlorophenyl Sulfide	1	2.5	5	12.5	20	30	40	50	BRC
n-Decane	1	2.5	5	12.5	20	30	40	50	HSL
n-Hydroxymethlphthalimide	1	2.5	5	12.5	20	30	40	50	HSL
Octachlorostyrene	1	2.5	5	12.5	20	30	40	50	BRC
p-Chlorophenyl Sulfone	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Disulfide	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Sulfide	1	2.5	5	12.5	20	30	40	50	BRC
Phenyl Mercaptan	1	2.5	5	12.5	20	30	40	50	REF
Phthalic Acid	1	2.5	5	12.5	20	30	40	50	REF
Phthalic Anhydride	1	2.5	5	12.5	20	30	40	50	REF
Quinoline	1	2.5	5	12.5	20	30	40	50	AP9

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APPENDIX A

Modifications Required for Analysis of Wastewater Following Method 625

REQUIREMENTS FOR METHOD 625

- Method 625 is required for demonstration of compliance with NPDES wastewater discharge permits or other CWA compliance situations. The standard analyte list and reporting limits are listed in Table A-1.
- This method can be applied to only aqueous matrices.
- The tune period for this method is defined as 24 hours. For 625.1 samples, the tune period is defined as 12 hours.
- Initial calibration curve requirements are as follows:
- The initial calibration curve for this method requires at least three points. 625.1 requires the use of at least 5 points.
  - Target compounds must have RSD $\leq$ 35%.
  - If this requirement cannot be met, a regression curve must be constructed for the non-compliant compounds.
- Continuing calibration verification requirements are as follows:
  - All target compounds must have %D $\leq$ 20%.
- Matrix Spike and LCS requirements are as follows:
  - A full analyte spike is required for method 625. See sections 7.8 and 7.9.
  - Method 625 requires a MS/D once per batch up to 20 samples.
  - Method 625 recommends field duplicates be analyzed for precision but there is no required precision requirement. In addition to matrix spikes the laboratory performs LCS spikes which provides long-term precision for the laboratory.
- IS and surrogate associations for target compounds are the same as Tables 6-8 for applicable compounds, as well as the characteristic masses used for quantitation and identification.

Table A-1.
TestAmerica Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Phenol	108-95-2	10
Bis(2-chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
1,2-Dichlorobenzene	95-50-1	10
2,2'-oxybis(1-chloropropane)	108-60-1	10
N-Nitroso-di-n-propylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Bis(2-chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
Hexachlorocyclopentadiene	77-47-4	20
2,4,6-Trichlorophenol	88-06-2	10
2-Chloronaphthalene	91-58-7	10
Dimethyl phthalate	131-11-3	10
Acenaphthylene	208-96-8	10
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50
2,4-Dinitrotoluene	121-14-2	10
2,6-Dinitrotoluene	606-20-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl phenyl ether	7005-72-3	10
Fluorene	86-73-7	10
4,6-Dinitro-2-methylphenol	534-52-1	50
N-Nitrosodiphenylamine	86-30-6	10
4-Bromophenyl phenyl ether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10

Table A-1. (continued)
TestAmerica Method 625 Standard Reporting List and Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
Di-n-butyl phthalate	84-74-2	10
Fluoranthene	206-44-0	10
Benzidine	92-87-5	100
Pyrene	129-00-0	10
Butyl benzyl phthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	50
Benzo(a)anthracene	56-55-3	10
Bis(2-ethylhexyl)phthalate	117-81-7	10
Chrysene	218-01-9	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10
N-Nitrosodimethylamine	62-75-9	10

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Table A-2.
Initial Demonstration Recovery and Precision Limits for Method 625

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Acenaphthene	60	27.6	60.1-132.3
Acenaphthylene	60	40.2	53.5-126.0
Aldrin[1]	60	39.0	7.2-152.2
Anthracene	60	32.0	43.4-118.0
Benz(a)anthracene	60	27.6	41.8-133.0
Benzo(b)fluoranthene	60	38.8	42.0-140.4
Benzo(k)fluoranthene	60	32.3	25.2-145.7
Benzo(a)pyrene	60	39.0	31.7-148.0
Benzo(ghi)perylene	60	58.9	D-195.0
Benzylbutyl phthalate	60	23.4	D-139.9
B-BHC[1]	60	31.5	41.5-130.6
d-BHC[1]	60	21.6	D-100.0
Bis(2-chloroethyl) ether	60	55.0	42.9-126.0
Bis(2-chloroethoxy)methane	60	34.5	49.2-164.7
Bis(2-chloroisopropyl) ether	60	46.3	62.8-138.6
Bis(2-ethylhexyl) phthalate	60	41.1	28.9-136.8
4-Bromophenyl phenyl ether	60	23.0	64.9-114.4
2-Chloronaphthalene	60	13.0	64.5-113.5
4-Chlorophenyl phenyl ether	60	33.4	38.4-144.7
Chrysene	60	48.3	44.1-139.9
4,4'-DDD[1]	60	31.0	D-134.5
4,4'-DDE[1]	60	32.0	19.2-119.7
4,4'-DDT[1]	60	61.6	D-170.6
Dibenzo(a,h)anthracene	60	70.0	D-199.7
Di-n-butyl phthalate	60	16.7	8.4-111.0
1,2-Dichlorobenzene	60	30.9	48.6-112.0
1,3-Dichlorobenzene	60	41.7	16.7-153.9
1,4-Dichlorobenzene	60	32.1	37.3-105.7
3,3'-Dichlorobenzidine	60	71.4	8.2-212.5
Dieldrin[1]	60	30.7	44.3-119.3
Diethyl phthalate	60	26.5	D-100.0
Dimethyl phthalate	60	23.2	D-100.0
2,4-Dinitrotoluene	60	21.8	47.5-126.9
2,6-Dinitrotoluene	60	29.6	68.1-136.7
Di-n-octylphthalate	60	31.4	18.6-131.8
Endosulfan sulfate[1]	60	16.7	D-103.5
Fluoranthene	60	32.8	42.9-121.3
Fluorene	60	20.7	71.6-108.4
Heptachlor[1]	60	37.2	D-172.2
Heptachlor epoxide[1]	60	54.7	70.9-109.4
Hexachlorobenzene	60	24.9	7.8-141.5
Hexachlorobutadiene	60	26.3	37.8-102.2
Hexachloroethane	60	24.5	55.2-100.0
Indeno(1,2,3-cd)pyrene	60	44.6	D-150.9

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Table A-2. (continued)
Initial Demonstration Recovery and Precision Limits for Method 625

Compound	Spiking Concentration, µg/L	Limit for Relative Standard Deviation	Limit for Average Recovery, %
Isophorone	60	63.3	46.6-180.2
Naphthalene	60	30.1	35.6-119.6
Nitrobenzene	60	39.3	54.3-157.6
N-Nitrosodi-n-propylamine	60	55.4	13.6-197.9
PCB-1260[1]	60	54.2	19.3-121.0
Phenanthrene	60	20.6	65.2-108.7
Pyrene	60	25.2	69.6-100.0
1,2,4-Trichlorobenzene	60	28.1	57.3-129.2
4-Chloro-3-methylphenol	60	37.2	40.8-127.9
2-Chlorophenol	60	28.7	36.2-120.4
2,4-Chlorophenol	60	26.4	52.5-121.7
2,4-Dimethylphenol	60	26.1	41.8-109.0
2,4-Dinitrophenol	60	49.8	D-172.9
2-Methyl-4,6-dinitrophenol	60	93.2	53.0-100.0
2-Nitrophenol	60	35.2	45.0-166.7
4-Nitrophenol	60	47.2	13.0-106.5
Pentachlorophenol	60	48.9	38.1-151.8
Phenol	60	22.6	16.6-100.0
2,4,6-Trichlorophenol	60	31.7	52.4-129.2

1. Organochlorine pesticides and PCBs project DQOs generally require better sensitivity than is provided by 8270C, so methods 8081 and 8082 are used instead. These compounds will not be included in the initial demonstration of capability for method 8270C.

APPENDIX B

Modifications Required for Analysis of Wastewater Following Method 8270 Best Practice (TALS Method Code 8270_LL)

REQUIREMENTS FOR METHOD 8270 BEST PRACTICE (8270_LL)

- The Best Practice Method is utilized to obtain lower reporting limits while still providing full scan data. The standard analyte list and reporting limits are listed in Table B-1.
- This method can be applied to only aqueous matrices.
- The extraction is the same except the final volume of the extract is 2 mL and there is an option for a Base-only extraction.
- The tune period for this method is defined as 12 hours.
- Initial calibration curve requirements are as follows:
  - Same as for 8270 detailed in Section 11.4 of this SOP.
  - The calibrations levels are shown in Table B-2.
- Continuing calibration verification requirements are as follows:
  - Same as for 8270 detailed in Section 11.5 of this SOP, except the level 7 calibration point (as defined in Table B-2) is used.
- Matrix Spike and LCS requirements are as follows:
  - The spike levels are listed in Table B-3.
- Internal Standards: The internal standard concentrations are listed in Table B-5.
- Surrogates: The surrogate concentrations are listed in Table B-4.
- Instrument Conditions are shown in Table B-6.
- Internal standard and surrogate associations for 8270_LL base-only extraction is shown in Table B-7. IS and surrogate associations for 8270_LL for the acid-base extraction are the same as Tables 6-8 for applicable compounds, as well as the characteristic masses used for quantitation and identification.

Table B-1.
TestAmerica Method 8270_LL Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
1,2,4-Trichlorobenzene	120-82-1	5
2,2'-oxybis(1-chloropropane)[2]	108-60-1	5
2,4,5-Trichlorophenol	95-95-4	5
2,4,6-Trichlorophenol	88-06-2	5
2,4-Dichlorophenol	120-83-2	5
2,4-Dinitrophenol	51-28-5	20
2,4-Dinitrotoluene	121-14-2	5
2,6-Dinitrotoluene	606-20-2	5
2-Chloronaphthalene	91-58-7	5
2-Chlorophenol	95-57-8	5
2-Methylnaphthalene	91-57-6	5
2-Methylphenol	95-48-7	5
2-Nitroaniline	88-74-4	5
2-Nitrophenol	88-75-5	5
3-Nitroaniline	99-09-2	5
4,6-Dinitro-2-methylphenol	534-52-1	10
4-Bromophenyl phenyl ether	101-55-3	5
4-Chloro-3-methylphenol	59-50-7	5
4-Chloroaniline	106-47-8	5
4-Chlorophenyl phenyl ether	7005-72-3	5
4-Methylphenol	106-44-5	5
4-Nitroaniline	100-01-6	5
4-Nitrophenol	100-02-7	5
Acenaphthene	83-32-9	5
Acenaphthylene	208-96-8	5
Aniline	62-53-3	5
Azobenzene	103-33-3	5
Benzoic acid	65-85-0	10
Benzyl alcohol	100-51-6	5
Bis(2-chloroethoxy)methane	111-91-1	5
Bis(2-chloroethyl)ether	111-44-4	1
Dibenzofuran	132-64-9	5
Dimethyl phthalate	131-11-3	5
Fluorene	86-73-7	1
Hexachlorobutadiene	87-68-3	5
Hexachlorocyclopentadiene	77-47-4	5
Hexachloroethane	67-72-1	5
Isophorone	78-59-1	5
Naphthalene	91-20-3	5
Nitrobenzene	98-95-3	5
N-Nitrosodimethylamine	62-75-9	1
N-Nitroso-di-n-propylamine	621-64-7	5
N-Nitrosodiphenylamine	86-30-6	5
Phenol	108-95-2	10
Pyridine	110-86-1	1.5

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Table B-1. (continued)
TestAmerica Method 8270_LL Standard Reporting Limits

Analytes	CAS Number	Aqueous, µg/L
1,4-Dioxane	123-91-2	1
3,3'-Dichlorobenzidine	91-94-1	5
Anthracene	120-12-7	5
Benzo(a)anthracene	56-55-3	1
Benzo(a)pyrene	50-32-8	5
Benzo(b)fluoranthene	205-99-2	5
Benzo(g,h,i)perylene	191-24-2	5
Benzo(k)fluoranthene	207-08-9	5
Bis(2-ethylhexyl)phthalate	117-81-7	5
Butyl benzyl phthalate	85-68-7	5
Carbazole	86-74-8	5
Chrysene	218-01-9	1
Dibenz(a,h)anthracene	53-70-3	5
Diethyl phthalate	84-66-2	5
Di-n-butyl phthalate	84-74-2	5
Di-n-octylphthalate	117-84-0	5
Fluoranthene	206-44-0	5
Hexachlorobenzene	118-74-1	1
Indeno(1,2,3-cd)pyrene	193-39-5	5
Pentachlorophenol	87-86-5	10
Phenanthrene	85-01-8	10
Pyrene	129-00-0	20

Additional compounds may be available upon request. Contact lab.

Table B-2.
Method 8270_LL Calibration Levels

Calibration Level	Calibration Concentration, µg/mL
1	0.25
2	0.50
3	1.00
4	2.50
5	5.00
6	7.50
7	10.
8	12.5
9	20.0
10	40.0
ICV	5.0 or 10.

Not all analytes are calibrated at all levels depending upon standard RL.

Table B-3.
Method 8270_LL LCS Spike Concentrations

LCS Compounds	Spiking Level, ug/L
Phenol	10
Bis(2-chloroethyl)ether	10
2-Chlorophenol	10
1,3-Dichlorobenzene	10
1,4-Dichlorobenzene	10
1,2-Dichlorobenzene	10
2,2'-oxybis(1-chloropropane)	10
N-Nitroso-di-n-propylamine	10
Hexachloroethane	10
Nitrobenzene	10
Isophorone	10
2-Nitrophenol	10
2,4-Dimethylphenol	10
Bis(2-chloroethoxy)methane	10
2,4-Dichlorophenol	10
1,2,4-Trichlorobenzene	10
Naphthalene	10
Hexachlorobutadiene	10
4-Chloro-3-methylphenol	10
Hexachlorocyclopentadiene	10
2,4,6-Trichlorophenol	10
2-Chloronaphthalene	10
Dimethyl phthalate	10
Acenaphthylene	10
Acenaphthene	10
2,4-Dinitrophenol	20
4-Nitrophenol	20
2,4-Dinitrotoluene	10
2,6-Dinitrotoluene	10
Diethylphthalate	10
4-Chlorophenyl phenyl ether	10
Fluorene	10
4,6-Dinitro-2-methylphenol	20
N-Nitrosodiphenylamine	10
4-Bromophenyl phenyl ether	10
Hexachlorobenzene	10
Pentachlorophenol	20
Phenanthrene	10
Anthracene	10
Di-n-butyl phthalate	10
Fluoranthene	10
Benzidine	10
Pyrene	10
Butyl benzyl phthalate	10
3,3'-Dichlorobenzidine	10
Benzo(a)anthracene	10

Table B-3. (continued)
Method 8270_LL LCS Spike Concentrations

LCS Compounds	Spiking Level, µg/L
Bis(2-ethylhexyl)phthalate	10
Chrysene	10
Di-n-octylphthalate	10
Benzo(b)fluoranthene	10
Benzo(k)fluoranthene	10
Benzo(a)pyrene	10
Indeno(1,2,3-cd)pyrene	10
Dibenz(a,h)anthracene	10
Benzo(g,h,i)perylene	10
N-Nitrosodimethylamine	10
1,4-Dioxane	10

Table B-4.
8270_LL Surrogate Compounds

Surrogate Compounds	Spiking Level, µg/L
Nitrobenzene-d_5	5
2-Fluorobiphenyl	5
Terphenyl-d_{14}	5
1,2-Dichlorobenzene-d_4[1]	5
Phenol-d_5	5
2-Fluorophenol	5
2,4,6-Tribromophenol	5
2-Chlorophenol-d_4[1]	5

[1] Included in standard mix, but not routinely evaluated for method 8270C

Recovery limits for surrogates are generated from historical data and are maintained by the QA department and stored in the LIMS

Table B-5.
8270_LL Internal Standard Compounds

Internal Standard Compounds	Spiking Level, ng/mL on-column
1,4-Dichlorobenzene-d_4	4000
Naphthalene-d_8	4000
Acenaphthene-d_{10}	4000
Phenanthrene-d_{10}	4000
Terphenyl-d_{14}	4000
Chrysene-d_{12}	4000
Perylene-d_{12}	4000

Table B-6.
Suggested Instrument Conditions for 8270_LL

Parameter	Value
Mass Range:	35 - 500 amu
Scan Time:	≤1 second/scan
Initial Column Temperature/Hold Time:	50 °C for 1 minute
Column Temperature Program:	50 - 320 °C at 35°C/min.
Final Column Temperature/Hold Time:	325 °C for 4 minute hold
Injector Temperature:	275 °C
Transfer Line Temperature:	290 °C
Source Temperature:	230 °C
Injector:	Single Taper Direct Connect Liner / Splitless injection
Injection Volume:	0.5 µL
Carrier Gas:	Helium at 1.0 mL/min.
Column:	DB-5 Capillary 20m x 0.18mm x 0.36 um film thickness

Table B-7.
Internal Standard and Surrogate Association for 8270_LL Base-Only Extraction
(in alpha-numeric order)

Analyte	IS	Surr
1,4-Dichlorobenzene-d_4 (Internal Standard)	1	-
1,4-Dioxane	**1**	**2**
2-Fluorobiphenyl (Surrogate Standard)	3	2
Acenaphthene-d_{10} (Internal Standard)	3	-
Chrysene-d_{12} (Internal Standard)	5	-
Naphthalene-d_8 (Internal Standard)	2	-
Nitrobenzene-d_5 (Surrogate Standard)	2	4
N-Nitrosodimethylamine	**1**	**2**
Perylene-d_{12} (Internal Standard)	6	-
Phenanthrene-d_{10} (Internal Standard)	4	-
Terphenyl-d_{14} (Surrogate Standard)	5	6

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APPENDIX C

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

MASS SPECTROMETER Instrument Maintenance Schedule				
Daily	**Weekly**	**As Needed**	**Quarterly**	**Annually**
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43).	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between service contract maintenance.	Check vacuum, relays, gas pressures, and flows.	Replace the exhaust filters on the mechanical rough pump every 1 to 2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.		Change the oil in the mechanical rough pump annually.
Check inlets, septa.		Clean source, including all ceramics and lenses. Source cleaning is indicated by a variety of symptoms, including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.		Relubricate the turbomolecular pump-bearing wick.
Check baseline level.		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates the need for replacement.		

APPENDIX C (continued)

Instrument Maintenance Schedules
Mass Spectrometer & Gas Chromatograph

GAS CHROMATOGRAPH Instrument Maintenance Schedule (For GC/MS only.)	
Daily	***As Needed***
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or guard column or break off front portion of capillary columns. Replace column if this fails to restore column performance or when column performance indicates it is required (e.g., peak tailing, poor resolution, high backgrounds, etc.).
Check temperatures of injectors and detectors. Verify temperature programs.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Check inlets, septa. Clean injector port.	Replace septa.
Check baseline level.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).
Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks.	Repair or replace flow controller if constant gas flow cannot be maintained.
	Reactivate flow controller filter dryers when the presence of moisture is suspected.
	Autosampler: Replace syringe, fill wash bottle, dispose of waste bottle contents.

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Inductively Coupled Plasma Mass Spectrometry for Trace Element Analysis by SW-846 Method 6020A/B

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1.0 Scope and Application

1.1 This procedure describes multi-elemental analysis by inductively coupled plasma-mass spectrometry (ICP-MS) based on EPA Method 6020A and 6020B.

1.2 Method 6020A and 6020B lists twenty-three elements approved for analysis by ICP-MS. The laboratory has implemented analysis by these methods for : Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mg, Mn, Ni, Se, Ag, Sr, Tl, Th, V, and Zn. Additional elements may be included provided that the method performance criteria presented in Sections 9 and 12 are met. Project approval may be required from the controlling agencies for compliance testing beyond the elements included in the promulgated methods and for those elements which may require state-specific accreditation.

1.3 The procedure is applicable to the analysis of acid digested waters, sediments, sludges and soils. Standard reporting limits are listed in Attachment 1 for water and soil. The preliminary acid digestion for aqueous samples is described in SOP DV-IP-0014 for Methods 3005A and 3020A and the digestion procedure for solids is given in SOP DV-IP-0015 for Method 3050B.

2.0 Summary of Method

2.1 Aqueous digestates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through the quartz torch and injects it into a radiofrequency (RF) plasma. There the sample is decomposed and desolvated.

2.2 The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrapole mass spectrometer capable of providing a mass resolution better than or equal to 0.9 amu (see Section 3) peak width at 10% of the peak height. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

2.3 A collision/reaction cell utilizing He and (optionally) H_2 gases is used to remove molecular interferences. As the ion beam passes through the cell chamber, a diffuse cloud of He or H_2 gas is injected into its path. Collisions between the ions and the atoms in the gas deflect and remove interferences. See Section 4.2.3 for more information.

2.4 Interferences must be assessed and valid corrections applied, or the data flagged to indicate problems. Interference corrections must include compensation for background ions contributed by the plasma gas, reagents, and the constituents of the sample matrix. Recommended elemental equations, which correct for many of these interferences, are listed in Attachment 2. Interference equations may vary or be unnecessary depending on the instrument setup and choice of collision/reaction gas.

2.5 Use of the internal standard technique is required to compensate for suppressions and enhancements caused by sample matrices. Internal standard assignments are listed in Attachment 4.

3.0 Definitions

3.1 **Atomic Mass Unit (amu) –** Obsolete term replaced by "unified atomic mass unit (u)" or "dalton (Da)", which denotes a small unit of mass that is used to express atomic and molecular masses. It is defined to be 1/12 of the mass of one atom of carbon-12.

3.2 **Batch –** The batch is a set of up to 20 samples of the same or similar matrix, plus required QC samples, processed using the same procedures and reagents within the same time period. Batches are defined at the sample preparation stage. See Policy DV-QA-003P for further details.

3.3 **Dissolved Metals** - Those elements which pass through a 0.45-µm membrane filter (sample is acidified after filtration).

3.4 **Total Metals** - The concentration determined on an unfiltered sample following vigorous acid digestion.

3.5 **Total Recoverable Metals** - The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acids.

3.6 **Instrument Detection Limit (IDL)** - See Section 12.3.

3.7 **Sensitivity** - The slope of the analytical curve (i.e., the functional relationship between raw instrument signal and the concentration).

3.8 **Tuning Solution** - This is a multi-element solution containing analytes which are representative of the entire mass range capable of being scanned by the instrument. It is used to optimize the sensitivity of the instrument and to verify the mass resolution meets method criteria.

3.9 **Initial Calibration Verification / Quality Control Standard (ICV)** - A multi-element standard of known concentrations prepared to verify instrument calibration. This solution must be an independent standard prepared near the mid-point of the calibration curve, and at a concentration other than that used for instrument calibration.

3.10 **Continuing Calibration Verification (CCV)** - A multi-element standard of known concentrations prepared to monitor and verify the instrument daily continuing performance.

3.11 **Interference Check Standard (ICS)** - A solution containing both interfering and analyte elements of known concentration that is used to correction factors.

3.12 **Laboratory Control Sample / Laboratory Fortified Blank (LCS/LFB)** - A multi-element standard of known concentrations that is carried through the entire sample preparation and analysis procedure. This solution is used to verify the accuracy of the sample preparation.

3.13 **Reagent Blank** - High purity (> 18 megohm-cm) water containing the same acid matrix as the calibration standards that is carried through the entire digestion

process.

3.14 **Calibration Blank** - High purity (> 18 megohm- cm) water acidified with the same acid concentrations present in the standards and samples. Also referred to as the Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB).

3.15 **Method Detection Limit (MDL)** - The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

3.16 **Low Level ICV (LLICV) / Continuing Calibration Verification (LLCCV)** - A multi-element standard of known concentrations prepared to monitor and verify the instrument performance at the reporting limit (RL).

3.17 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

4.1 Elemental Isobaric Interferences

Elemental isobaric interferences in the ICPMS are caused by isotopes of different elements forming ions with the same nominal mass-to-charge ratio (m/z). Most interferences of this type are corrected for by the instrument software and by the careful selection of isotopes for analysis.

4.2 Isobaric Molecular Interferences

4.2.1 Polyatomic interferences are derived from the plasma gas, reagents or sample matrix. Isobaric molecular interferences are caused by ions consisting of more than one atom or charge. Attachment 3 lists isobaric interferences which might possibly affect required analytes. These molecular interferences are minimized by use of the collision cell utilizing He and/or H_2 gases. When these interferences cannot be avoided by the use of another isotope with sufficient natural abundance, corrections must be applied and the data flagged to indicate the presence of interferences.

4.2.2 Chloride in samples can produce low recoveries for antimony and silver. If chloride interference is a concern, 1% HCl can be added during digestion, but calibration standards must be adjusted to include 1% HCl also. The use of hydrochloric and sulfuric acids should be minimized due to higher incidence of molecular-ion interferences with the presence of these acids. Excessive amounts of nitric acid can also lead to molecular interferences.

4.2.3 Collision cell interference removal works both by causing the interfering molecular ion to dissociate and by reducing the kinetic energy of the ion. The latter is termed Kinetic Energy Discrimination (KED), and is the

primary mechanism for interference removal. Polyatomic ions are larger than elemental ions and so collide with the helium atoms in the collision cell more frequently than the smaller elemental ions. Each collision reduces the energy of the ion, so the molecular ions lose energy more quickly. At the end of the collision cell a positive voltage plate prevents passage of the now low energy molecular ions. Thus, the interference is eliminated because the molecular ions do not reach the detector.

4.3 Doubly Charged Ion Interferences

Doubly charged elemental ion interferences are possible in cases where the second ionization potential of the element is significantly below the first ionization potential for argon (15.7 eV). If a doubly charged ion is formed, it will cause a response at half of its elemental mass, potentially causing interference. Most elements have high enough second ionization potentials that formation of doubly charged ions is not an issue. The percentage of doubly charged ions being formed in the plasma is monitored on a daily basis during the instrument tuning process.

4.4 Physical Interferences

4.4.1 Physical interferences are associated with the transport and nebulization process. Internal standards are used to compensate for these types of interferences.

4.4.2 Internal standards should be added at a level to give approximately 100,000 – 20,000,000 counts of raw signal intensity. The mass of the internal standard should ideally be within 50 amu of the mass of the measured analyte.

4.4.3 Matrix effects are monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the calibration blank. When performing method 6020A, the internal standard recoveries in samples can not fall below 70% of the intensity of the calibration standard. For method 6020B, the internal standard recoveries in samples cannot fall below 30% while the requirement for DoD is 30-120%. If they fall outside the applicable window, a five-fold dilution (1:4) is performed on the sample to correct for matrix effects and the sample is reanalyzed.

4.4.4 Memory effects or carry-over can occur when there are large relative concentration differences between samples and/or standards which are analyzed sequentially. The rinse period between samples must be long enough to eliminate significant memory interference.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately.

5.3.2 The ICP-MS plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma. The RF Generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section.

Material[1]	Hazards	Exposure Limit[2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

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6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) capable of providing resolution less than or equal to 0.9 amu at 10% peak height and 1.0 amu at 5% peak height in the mass range from 6-253 with a data system that allows corrections for isobaric interferences and the application of the internal standard technique. The ICP-MS must be equipped with a collision cell for the removal of molecular interferences.

6.1.2 A four-channel peristaltic pump.

6.1.3 Autosampler with autosampler tubes.

6.1.4 Appropriate water cooling device.

6.2 Supplies

6.2.1 Argon gas: High purity grade (99.99%).

6.2.2 Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.2.3 Class A volumetric flasks and 100ml Graduated Cylinder

6.3 Computer Software and Hardware

Please refer to the master list of documents and software located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. All standards must be entered into the TestAmerica LIMS (TALS) Reagent Module. Reagents that are not used for calculating results may either be recorded in the Reagent Module or may be entered into batch worksheets.

7.1 Storage and Shelf-Life

7.1.1 All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Standards stored at concentrations as received from the vendor and mid-level dilutions must be replaced prior to the expiration date assigned by the vendor. If no expiration date is provided, the stocks and mid-level standards may be stored for up to one

year. They must be replaced sooner if verification from an independent source indicates a problem.

7.1.2 Working standards, i.e., all standards at concentrations ready to analyze on the ICP-MS are prepared fresh daily.

7.1.3 For more information on standard storage and shelf-life, see SOP DV-QA-0015.

7.2 Standards

Detailed instructions regarding the preparation of standards and reagents are given in this section. Alternate procedures are allowed as necessary to accommodate volume requirements as long as final concentrations are maintained and an accurate description of the standard or reagent used is entered into the Reagent Module in TALS.

7.2.1 Tuning Solution

The parent tuning solution is purchased as a custom multi-element mix. The elements and concentrations of the constituents are shown in Attachment 7. Prepare the working tuning Solution as detailed below.

7.2.1.1 Obtain a clean 1 L volumetric flask

7.2.1.2 Place 500 mL of reagent water and 10 mL of conc. HNO3 in the flask

7.2.1.3 Pipette 1mL of the Tuning Solution Stock into the flask

7.2.1.4 Add 50 µL of a 500 mg/L Mg solution and 30 µL of a 1000 mg/L Be solution.

7.2.1.5 Dilute to volume with reagent blank (See Section 7.3). Stopper and mix.

7.2.2 P/A factor solution

7.2.2.1 The Pulse/Analog (P/A) solution is used to monitor the correlation between the Pulse counting and Analog modes of the electron multiplier. The diluted solution must be prepared at different concentrations depending on the current instrument conditions. Multiple dilutions may be required to cover the required intensity range for all elements.

7.2.2.2 The P/A solution may be commercially purchased as a custom multi-element mix. See Attachment 7 for a list of the constituents and concentrations.

7.2.2.3 Prepare and use the P/A solution as recommended by the instrument manufacturer. The P/A solution should be analyzed daily.

7.2.3 Calibration Standard

Stock calibration standards are purchased as custom multi-element mixes or as single element solutions. Each day of analysis, the standards are diluted to working levels using reagent blank (see Section 7.3). The concentrations are given in Attachment 10. Prepare the Daily Working Calibration Standard as shown.

7.2.3.1 Daily Working Calibration Standard for Instruments 077 and 078 (ms 77 cal std)

7.2.3.1.1 Obtain a clean 200 mL volumetric flask.

7.2.3.1.2 Place 100 mL of reagent blank in the flask.

7.2.3.1.3 Pipette 0.25 ml of each MS CALSTD 1 thru 6 into the flask.

7.2.3.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.4 Initial Calibration Verification (ICV) Standard

The ICV stock is from a source different than the source for the calibration standards. Each day of analysis, the ICV standards are prepared new in reagent blank to the concentrations shown in Attachment 10. Prepare the ICV as shown below:

7.2.4.1 Initial Calibration Verification Standard for Instruments 077 and 078 (MS 77 ICV)

7.2.4.1.1 Obtain a clean 250 mL volumetric flask.

7.2.4.1.2 Place 100 mL of reagent blank in the flask.

7.2.4.1.3 Pipette 0.1 mL of each MS ICVMIX 1 thru 6 into the flask.

7.2.4.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.5 Continuing Calibration Verification (CCV) Standard

The CCV is prepared from the same source as the calibration standards. The CCV standards are prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10.

7.2.5.1 Continuing Calibration Verification for Instruments 077 and 078 (ms 77 ccv)

7.2.5.1.1 Using a graduated cylinder pour 100 ml each of the MS CAL Daily and reagent blank for a final volume of 200 ml.

7.2.6 Reporting Limit (RL) Standards

The reporting limit standards are prepared fresh daily from the same stock as the calibration standards using the reagent blank. The analyte concentrations must be less than or equal to the respective reporting limits. Multiple solutions may be required in order to satisfy all of the project and client specific reporting limits. Alternate reporting limit concentrations may be used as necessary to meet client requirements as long as an accurate description of the standard used is entered into the Reagents module in TALS. Prepare the Reporting Limit Standard for the Agilent 7700 as detailed below.

7.2.6.1 RL Standard for Instruments 077 and 078 (ms 77 RL)

7.2.6.1.1 Obtain a clean 50 mL volumetric flask.

7.2.6.1.2 Place 30 mL of reagent blank in the flask.

7.2.6.1.3 Pipette 0.5 mL of the ms 77 cal std solution into the flask.

7.2.6.1.4 Dilute to volume with reagent blank. Stopper and mix.

7.2.7 Daily Linear Range Standard

The Linear Range standard is prepared from the same stock as the calibration standards using reagent blank.

7.2.7.1 Daily Linear Range Standard for Instruments 077 and 078 (MS 77 LR STD)

7.2.7.1.1 Obtain a clean 500 mL volumetric flask.

7.2.7.1.2 Place 50 mL of reagent blank in the flask.

7.2.7.1.3 Pipette 1.0 mL of a 1,000 mg/L As standard into the flask.

7.2.7.1.4 Pipette 2.5 mL of a 1,000 mg/L Ba standard into the flask.

7.2.7.1.5 Pipette 1.0 mL of a 1,000 mg/L Be standard into the flask.

7.2.7.1.6 Pipette 1.0 mL of a 1,000 mg/L Cd standard into the flask.

7.2.7.1.7 Pipette 1.0 mL of a 1,000 mg/L Co standard into the flask.

7.2.7.1.8 Pipette 2.5 mL of a 1,000 mg/L Cr standard into the flask.

7.2.7.1.9 Pipette 2.5 mL of a 1,000 mg/L Cu standard into the flask.

7.2.7.1.10 Pipette 1.0 mL of a 1,000 mg/l Li standard into the flask.

7.2.7.1.11 Pipette 5.0 mL of a 1,000 mg/L Mn standard into the flask.

7.2.7.1.12 Pipette 1.0 mL of a 1,000 mg/L Mo standard into the flask.

7.2.7.1.13 Pipette 2.5 mL of a 1,000 mg/L Ni standard into the flask.

7.2.7.1.14 Pipette 2.5 mL of a 1,000 mg/L Pb standard into the flask.

7.2.7.1.15 Pipette 0.5 mL of a 1,000 mg/L Sb standard into the flask.

7.2.7.1.16 Pipette 1.0 mL of a 1,000 mg/L Se standard into the flask.

7.2.7.1.17 Pipette 1.0 mL of a 1,000 mg/L Sn standard into the flask.

7.2.7.1.18 Pipette 1.0 mL of a 1,000mg/l Sr standard into the flask.

7.2.7.1.19 Pipette 0.5 mL of a 1,000 mg/L Tl standard into the flask.

7.2.7.1.20 Pipette 1.0 mL of a 1,000 mg/L U standard into the flask.

7.2.7.1.21 Pipette 1.0 mL of a 1,000 mg/L V standard into the flask.

7.2.7.1.22 Pipette 2.5 mL of a 1,000 mg/L Zn standard into the flask.

7.2.7.1.23 Pipette 0.05 mL of a 10,000 mg/L Th standard into the flask.

7.2.7.1.24 Dilute to volume with reagent blank. Stopper and mix.

7.2.8 Internal Standard (IS) Solution (77 I.S. / MS I.S. INT)

The internal standard solution is added continuously by peristaltic pump through a mixing tee. The concentrations and components are specified in Attachment 4. Prepare the IS solution as follows:

7.2.8.1 Obtain a clean 250 mL volumetric flask.

7.2.8.2 Place 100 mL of reagent blank in the flask.

7.2.8.3 Pipette 1.2 mL of the 1,000 mg/L Ge Standard into the flask.

7.2.8.4 Pipette 0.4 mL of the 1,000 mg/L Ho Standard into the flask.

7.2.8.5 Pipette 0.4 mL of the 1,000 mg/L In Standard into the flask.

7.2.8.6 Pipette 0.75 mL of the 1,000 mg/L Sc Standard into the flask.

7.2.8.7 Pipette 1.5 mL of the 1,000 mg/L ^{6}Li Standard into the flask.

7.2.8.8 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9 Interference Check Standard Solutions (ICSA / ICSAB)

The interference check standard solution (ICSA) and the spiked interference check standard solution (ICSAB) are prepared as follows:

7.2.9.1 ICSA Standard (ms 77 icsa / MS ICSA)

7.2.9.1.1 Obtain a clean 100 mL volumetric flask.

7.2.9.1.2 Place 50 mL of reagent blank in the flask.

7.2.9.1.3 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.1.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.9.2 ICSAB Standard (MS 77 ICSAB / MS ICSAB)

7.2.9.2.1 Obtain a clean 100 mL volumetric flask.

7.2.9.2.2 Place 50 mL of reagent blank in the flask.

7.2.9.2.3 Pipette 0.1 mL of each MS ICVMIX 1 thru 6 stock standards into the flask.

7.2.9.2.4 Pipette 10.0 mL of the MS ICSA STOCK standard into the flask.

7.2.9.2.5 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.2.10 6020A only -Low Level Initial Calibration Verification and Low-Level Continuing Verifications (ms 77 LLCCV / MS LCCV)

The low level ICV / low level CCV solution is prepared from the same source as the calibration standards. The low level standard is prepared fresh each day of analysis in reagent blank. The concentration is shown in Attachment 10. Prepare the low level standard solution as follows:

7.2.10.1 Obtain a 100 mL volumetric flask.

7.2.10.2 Place 50 mL of reagent blank in the flask.

7.2.10.3 Pipette 1 ml of MS LLCCV

7.2.10.4 Dilute to volume with the appropriate reagent blank. Stopper and mix.

7.3 Reagents

7.3.1 Reagent Water – Water free of the elements of interest, generated using an ion-exchange water polishing system capable of achieving 18.0 megohm-cm.

7.3.2 Reagent Blank - Agilent 7700, 2% HNO3/0.5% HCl – Carefully dilute 40 mL of concentrated HNO3 and 10 mL of HCl in 2.0 L of reagent water. This solution is used to dilute samples and it is used for the initial and continuing calibration blanks.

7.3.3 Reagent Blank - Agilent 7700, 5% HNO3/5% HCL (Zr only) – Carefully dilute 100 ml of concentrated HNO_3 and 100 ml of HCL in 2.0 L of reagent water. This solution is used to dilute samples and it is used for calibration blanks.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time[1]	Reference
Water[2]	HDPE	50 mLs	HNO_3, pH < 2	180 Days	SW-846

Soil	Glass	4 oz	Cool ≤ 6°C[3]	180 Days	SW-846

[1]Samples must be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still considered valid. Holding Times are calculated from the date the sample was collected.

[2]Water samples collected for dissolved elements are filtered immediately on-site by the sampler before adding preservative.

[3]Although ICP analysis of soil does not require refrigeration of the samples, mercury analysis does require refrigeration per SW-846. Samples which will be used to aliquot for both analyses must be refrigerated. Therefore the laboratory routinely refrigerates samples to be analyzed by Methods 6020A or 6020B.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TALS Method Comments to determine specific QC requirements that apply. Quality control requirements are summarized in Attachment 9.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Method Blank (MB)

For aqueous and soil samples, the method blank consists of reagent water that has been processed in the same manner as the samples. For soil samples analyzed

under DoD QAPPs, the method blank consists of <1 mm glass beads that have been processed in the same manner as the samples. One method blank must be processed with each preparation batch.

Acceptance Criteria: Method blank results are acceptable if the concentration for each analyte of interest is less than ½ the reporting limit (RL). For DoD QSM 5.0 the control limit is less than ½ LOQ. In the absence of project specific reporting limits, if the blank is less than 10% of the lower limit of quantitation check sample concentration, less than 10% of the regulatory limit, or less than 10% of the lowest sample concentration for each analyte in a given preparation batch, whichever is greater, then the method blank is considered acceptable.

Corrective Action: If the method blank does not meet the acceptance criteria, the source of contamination should be investigated to determine if the problem can be minimized or eliminated. Samples associated with the contaminated blank shall be reprocessed for analysis or, under the following circumstances, may be reported as qualified (qualifier flags or narrative comments):

- The same analyte was not detected in the associated samples;
- The method blank concentration is less than 1/10 of the measured concentration of any sample in the batch;
- The method blank concentration is less than 1/10 the specified regulatory limit; or
- The analyte is a common laboratory contaminant (e.g., copper, zinc, iron, or lead) less than 2 times the RL. Note that some programs do not recognize common lab contaminants or have a more stringent criterion (e.g., DoD QSM 5.0 allows common laboratory contaminants up to the RL).

If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. This anomaly must be addressed in the project narrative and the client must be notified.

9.3 Laboratory Control Sample (LCS)

The LCS consists of reagent water that is spiked with the analytes of interest at the project specific action level or, when lacking specific action levels, at approximately the mid-point of the calibration range (summarized in Attachment 10). For soil samples analyzed under DoD QAPPs, the LCS consists of <1 mm glass beads that have been spiked with the analytes of interest and processed in the same manner as the samples. One LCS must be processed for each preparation batch.

Acceptance Criteria: LCS control limits are based on three standard deviations of past laboratory results or program specific requirements. These limits must not exceed 80-120%. The control limits are maintained in TALS. For DoD QSM 5.0 the laboratory must use QSM Appendix C Limits for batch control if project limits are not specified.

Corrective Action: If the LCS recovery falls outside of the control limits for any analyte, that analyte is judged to be out of control. All associated samples must be reanalyzed One possible exception is a recovery for a given element above the upper control limit with no detection for the same element in the samples. If project requirements allow this exception, the data may be accepted with qualifiers, an NCM must be generated, and the failure narrated in the final report.

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9.4 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

The MS is prepared by taking a second aliquot of a selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). An MSD is prepared by taking a third aliquot of the selected sample and spiking it with the analytes of interest at the same level as the LCS (summarized in Attachment 10). The MS and MSD are processed in the same manner as the samples. One MS/MSD pair must be processed for each preparation batch. Some programs (e.g., DoD) require that matrix spikes can be performed only on project samples, and that the samples to be used are identified on the chain of custody form. The spike concentration should be the same level as the LCS.

Acceptance Criteria: Control limits are based on historical data or project specific requirements. Historical control limits are based on three standard deviations of past laboratory results. These limits are not to exceed 75-125% recovery, and 20% relative percent difference (RPD). The control limits are maintained in TALS. For DoD QSM 5.0 the laboratory must use QSM Appendix C limits for batch control if project limits are not specified.

Corrective Actions: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the

results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as "NC" (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as "NC" (not calculated).

- For MS/MSD that serve as batch QC, if the parent sample result is within the calibration range and the MS/MSD results are above the calibration range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within calibration range and the MS/MSD results are above the calibration range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.
- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

NOTE: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE: This method does not require a sample duplicate. Precision is measured using the MS/MSD. Use of the MS/MSD is preferred as not all samples will contain measurable concentrations of the target analytes. Samples that have target analytes at low concentrations or non-detectable levels do not provide useful precision data. When an MS/MSD is not available, an LCS and LCSD will be used to measure precision.

9.5 Interference Check Solutions (ICSA/ICSAB)

The interference check solution is prepared with known concentrations of interfering elements so a determination may be made as to the magnitude of the interference on analytes of interest as well as a test of any software corrections. The required elements and their concentrations are listed in Attachment 5. The interference check solutions must be analyzed at the beginning of every analytical run and once every 12 hours thereafter. The results of solution "A" and solution "AB" should be monitored for possible interferences.

Acceptance Criteria: The non-spiked analytes in the A solution must be less than 2x the RL. The results for the trace elements (B portion) must be ± 20% of the expected value. In addition, the internal standard recoveries for both the ICSA and AB must be within 70-150% for Method 6020A, 30-150% for Method 6020B and 30-120% for DoD. Some programs have control limits for the non-spiked elements in the ICSA. Please check the client specific requirements. For DoD QSM 5.0 the ICSA for non-spiked elements is controlled to less than the absolute value of the LOD unless they are a verified impurity.

Corrective Action: If the ICSAB results exceed the 20% limit or the ICSA is out for DoD QSM 5.0, then the analysis sequence must be terminated. For DoD QSM 5.0 if the ICSA is outside of the control limits for the non-spiked elements the sequence must also be terminated. The problem must be investigated and fixed. The ICSA and all affected samples must be re-analyzed.

NOTE: It may not be possible to obtain absolutely clean ICSA/ICSAB standards. If contamination can be confirmed by another method (e.g., ICPAES), acceptance criteria will be applied at that level and the data accepted.

9.6 Internal Standards Evaluation for Samples

The IS recovery in samples cannot fall below 70% or be above 150% of the intensity of the calibration blank for 6020A and 30-150% for 6020B. If sample IS recoveries fall outside of these criteria, a five-fold (1:4) dilution must be performed, the dilution analyzed, and the same acceptance criteria applied. For DoD QSM 5.0 the internal standard for samples is controlled to 30-120%.

9.7 Serial Dilution

One serial five-fold dilution should be analyzed per preparation batch. If the analyte concentration is within the linear range of the instrument and sufficiently high (minimally, a factor of 50 times above the MDL), the serial dilution must agree to within 10% of the original analysis. If not, an interference effect is suspected, which must be described in an NCM and included in the final report narrative. Samples identified as blanks should not be used for serial dilution. For DoD QSM 5.0 the

serial dilution is evaluated if the parent sample concentration is greater than 50x the LOQ prior to dilution. If the acceptance criteria are not met then the parent sample is flagged "J". Method 6020B sets the calculation level at 25x RL and the required limit at 20%.

9.8 Post-Digestion Spike Addition (PDS)

A PDS is performed for each batch. An analytical spike added to a portion of a prepared sample, or its dilution, should be recovered to within 80 - 120% of the known value. If the PDS fails to meet this criterion, matrix interference should be suspected. Typically the concentration of the PDS is 200 µg/L for each element except silver which is spiked at 50 µg/L. For DoD QSM 5.0 if the parent sample concentration is less than 50x the LOQ prior to dilution then the PDS must recover within 80-120%. If the recovery is outside of the control limits for a given element then the parent sample is flagged "J". Method 6020B allows limits of ± 25%.

9.9 Linear Range Verification (LRA/LRC)

The LDRs should be verified whenever, in the judgment of the analyst, a change in the analytical performance caused by either a change in instrument hardware or operating conditions would dictate the necessity to re-establish them. 6020B and DoD QSM require verification of linear ranges in each analytical run. As described in Section 7.2.7, a lower concentration is used for the daily check than is used for the quarterly determination.

Acceptance Criteria: The result for this standard must be within 10% of the expected value.

Corrective Action: If the Linear Range Verification standard fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analyst must run a standard at a lower concentration until the criteria is met or the samples cannot exceed the level of the highest calibration standard.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any unauthorized deviations from this procedure identified after the work has been completed must also be documented in an NCM, with a cause and corrective action described.

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10.3 Instrument Maintenance

See Section 20 in the QAM

10.4 Instrument Troubleshooting

See Attachment 11

10.5 Sample Preparation

Solid and aqueous samples must be digested prior to analysis by the appropriate method (see SOPs DV-IP-0014 and DV-IP-0015).

10.6 Calibration

10.6.1 Instrument Start Up

Set up the instrument according to manufacturer's operating instructions. Allow the instrument to become thermally stable for at least 30 minutes before tuning. It is recommended that the instrument be flushed with the ICSA solution to help condition the cones and improve stability. Allow the instrument time to rinse completely before tuning the instrument.

10.6.2 Oxide\Doubly Charge Performance Check

With the sample probe in the Tune solution verify that the oxides and doubly charged ions are less the 3% by running the Tune report.

10.6.3 Instrument Tuning / Mass Calibration

Tune the instrument with a solution containing elements representing all of the mass regions of interest. The relative standard deviations must be less than 5% after running the tuning solution a minimum of 5 times. Mass calibration and resolution checks using the tuning solution must be completed at the beginning of every day. If either of the following conditions fails the instrument setup must be re-evaluated and the solution rerun.

Mass Calibration Check – The mass calibration results must be within 0.1 amu from the true value. If this criterion is not met, the mass calibration must be adjusted before running samples.

Mass Resolution Check - The resolution at 5% peak height should be approximately 0.75amu.

NOTE: Method 6020B states to use the manufacturer's instructions for the tune. Since the laboratory may perform analysis for Method 6020A on the same instrument, the same requirements are applied to both.

10.6.4 Initial Calibration

The ICP-MS is calibrated each day of operation using a blank and a single standard (see Section 7.2.3). At least three integrations are employed. The validity of the calibration is determined by the subsequent calibration verifications, which are performed at concentrations as described in the next sections.

10.6.5 Low-Level Initial Calibration Verification (LLICV/ICVL)

A low-level ICV standard at or below the reporting limit (see Section 7.2.10) is analyzed after the initial calibration for Method 6020A. This is a standard obtained from the same vendor used for calibration.

Acceptance Criteria: The ICVL recovery must be within 70-130%.

The ICVL can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICVL results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.6 Mid-Level Second-Source Initial Calibration Verification (ICV)

A 40 µg/L ICV standard (see Section 7.2.4) is analyzed immediately after the initial calibration. This is a standard obtained from a different vendor than the standard used for calibration.

Acceptance Criteria: The ICV recovery must be within 90-110%. The ICV can be reanalyzed, but two consecutive successful results must be obtained or corrective action is taken.

Corrective Action: If the ICV results are outside of the acceptance limits, investigate the accuracy of the standards, correct as necessary, and recalibrate.

10.6.7 Calibration Blank

An initial calibration blank (ICB) is analyzed after the ICV. Continuing calibration blanks (CCBs) are analyzed after each continuing calibration verification. The appropriate reagent blank is used for the blanks.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Common lab contaminants such as zinc and iron must be less than the RL. In addition, the internal standard recoveries must be within 70-150% of the associated calibration blank for Method 8020A or

30-150% for Method 6020B. Client specific requirements take precedence. DoD QSM 5.0 requires control of blanks to a concentration less than or equal to the LOD with the internal standard recoveries of 30-120%.

Corrective Action: If the calibration blank exceeds acceptance limits, then the possibility of instrument contamination should be examined, particularly the possibility of carry-over from high level samples. The blank can be reanalyzed, and if successful, analysis can continue. However, samples tested after high-level samples should be retested. If the reanalysis is not successful, then the analysis should be terminated. After the problem is corrected, recalibrate and reanalyze all samples tested since the last acceptable CCB.

10.6.8 Reporting Limit (RL/CRI) Verification Standard

Because the ICP-MS calibration does not include multiple calibration levels, an independent standard is analyzed after the ICV to monitor the lab's ability to produce reliable results at RL-level concentrations. The RL verification standard (see Section 7.2.6) is analyzed after the daily ICB.

Acceptance Criteria: For Method 6020A, the results should be within 50% of the expected value. Some programs may require tighter controls. For Method 6020B and DoD QSM 5.0 the control limits are 80-120%.

Corrective Action: If the RL verification fails to meet acceptance limits, data for the associated samples must be assessed. For example, if the results are high, consider blank contamination, and if the results are low, consider MDL verifications. At a minimum, sample results must be qualified in the final report. For DoD QSM 5.0, if the low-level standard does not meet the limits when spiked at the required project RL, the run sequence must be terminated.

10.6.9 Lower Limit of Quantitation Check (LLQC, LLOQ)

The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits, quarterly and on an as needed basis to demonstrate the desired detection capability. The difference between the LLQC and the RL is that this standard is carried through the entire preparation and analytical procedure. Prepare 7 aliqouts.

Acceptance Criteria: LLQC is verified when all analytes are detected within ± 35% of their true value. The RSD should be ≤20%

Corrective Action: If the LLQC fails to meet acceptance limits, a single reanalysis may be attempted without modification to the instrument operating conditions. Otherwise, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

10.6.10 6020A Only - Low-Continuing Calibration Verification (LLCCV/CCVL) Standard

A low-level CCV standard is analyzed after every set of ten samples and at the end of the analytical sequence.

Acceptance Criteria: The CCVL recovery must be within 70-130%. In addition, the IS recovery must be within method limits. If CCVL results are not within these limits, the CCVL can be reanalyzed, but it must be successful twice in succession. If the calibration cannot be verified within these specified limits, the analysis of samples containing the affected analytes at similar concentrations cannot continue until the cause is determined and the CCVL standard successfully analyzed.

For the state of Washington the CCVL must work on the first attempt.

Corrective Action: If the CCVL fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCVL. If the associated samples are at levels greater than 10X the level of the CCVL the data may be considered acceptable but the failure must be documented with an NCM and addressed in the case narrative.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCVL's following the failure are successful but the samples must still be rerun.

10.6.11 Continuing Calibration Verification (CCV) Standard

A 50 µg/L CCV standard (see Section 7.2.5) is analyzed after every set of ten samples or every 2 hours, whichever is most frequent, and at the end of the analytical sequence.

Acceptance Criteria: The CCV recovery must be within 90-110%. In addition, the IS recovery must be within 70-150% for Method 6020A or 30-150% for Method 6020B. If the CCV results are not within these limits, the CCV can be reanalyzed, but it must be successful twice in succession or further corrective action must be taken.

For the state of Washington the CCV must work on the first attempt

Corrective Action: If the CCV fails acceptance criteria, then the analysis should be terminated. Recalibrate and reanalyze all samples tested since the last acceptable CCV.

For the state of Washington the previous 10 samples must be reanalyzed. A recalibration is not necessary if the two CCV's following the failure are successful but the samples must still be rerun.

10.7 Sample Analysis

10.7.1 Report the average of at least three integrations for all field and QC samples analyzed.

10.7.2 Flush the system with the rinse blank for at least 30 seconds between samples and standards during the analytical run.

10.7.3 Masses which would affect the data quality must be monitored during the analytical run to determine the potential effects of matrix on a given element. See Attachment 3 for examples.

10.7.4 Dilute and reanalyze samples that are more concentrated than the linear range for an analyte. DoD QSM 5.0 requires that samples be diluted and reanalyzed if they are above the daily linear range check standard. No analyte may be reported from an analysis of a diluted sample in which the analyte concentration is less than 5 times the RL. (The sample should be diluted to the approximate midrange of the analytical curve.) See Section 9.9 for the linear range verification requirements.

10.7.5 The analytical run sequence should be performed as follows to meet all quality control criteria:

Instrument initialization / Warm-Up
Tune instrument
Perform mass calibration
Perform resolution check
Validate tuning criteria
Calibration blank

Calibration standard
ICV
ICB
LLICV
RL verification standard
LLQC(as needed)
ICSA
ICSAB
LRA
CCV
CCB
LLCCV (6020B)
10 Samples (which can include all sample types)
CCV
CCB
LLCCV (6020B)
Reslope
CCV
CCB
LLCCV

11.0 Calculations / Data Reduction

11.1 Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.2 ICV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{ICV Found Value}}{\text{ICV True Value}}\right) \times 100\%$$

11.3 CCV percent recoveries are calculated according to the equation:

$$\%R = \left(\frac{\text{CCV Found Value}}{\text{CCV True Value}}\right) \times 100\%$$

11.4 Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = \left(\frac{\text{SSR - SR}}{\text{SA}}\right) \times 100\%$$

Where:

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

NOTE: When the sample concentration is less than the detection limit, use SR = 0 for the purpose of calculating %R.

11.5 The relative percent difference (RPD) between sample duplicates is calculated according to the following equation:

$$RPD = \left[\frac{DU1 - DU2}{\frac{1}{2}(DU1 + DU2)} \right] \times 100$$

Where:

$DU1$ = Sample result
$DU2$ = Sample duplicate result

11.6 The final concentration for an aqueous sample is calculated as follows:

$$\text{Result } (\mu\text{g/L}) = \frac{C \times V1 \times D}{V2}$$

Where:

C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

11.7 The concentration determined in digested solid samples when reported on a dry weight basis is as follows:

$$\text{Result } (\mu\text{g/kg}) = \frac{C \times V \times D}{W \times S}$$

Where:

C = Concentration from instrument readout, ppb
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight, in g, of wet sample digested
S = Percent solids/100

11.8 Sample data are reviewed by the analyst (Level 1 data review) and documented on the data review checklist (See SOP DV-QA-0020). The data package is then submitted for level 2 review by another analyst or data reviewer. Second level

review is documented on the same checklist initiated by the analyst. The data review process is explained in SOP DV-QA-0020.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-002. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. For DoD, DOE and TX TRPP projects, an MDL verification is performed quarterly.

12.2 MDL Verification (MDLV)

Calculated MDLs from the annual studies are subject to quarterly verification by analyzing an MDLV standard.

12.2.1 Prepare an MDLV standard at 2-4 times the calculated MDL concentration.

12.2.2 Analyze the MDLV standard immediately after each MDL study and quarterly thereafter. This standard is subject to the entire preparation and analysis process.

12.2.3 The calculated MDL is verified if the MDLV standard is detected, nominally signal to noise ratio > 3, under routine instrument conditions.

12.2.4 If the first MDLV is not detected, re-prepare the MDLV standard at twice the original concentration and analyze. The lowest concentration that produces a detectable signal will then be reported as the MDL.

12.3 Instrument Detection Limit Study

Instrument detection limit (IDL) studies are conducted quarterly for each instrument and each analyte used for analysis in accordance with Policy DV-QA-014.

12.3.1 Pour out seven undigested calibration blanks and run them on three non-consecutive days.

12.3.2 Calculate the standard deviation for each day. The final IDL concentration is the average of the three daily standard deviation values.

12.3.3 Method 6020B requires an initial verification of the IDL using 10 replicates in a single analytical sequence but no longer requires quarterly

verification. Reverification is required after major maintenance, such as changing the detector.

12.3.4 See Policy DV-QA-014P for a discussion of IDL studies and evaluation of IDL results.

12.4 Linear Dynamic Range (LDR)

12.4.1 The LDR must be determined initially (i.e., at initial setup) and then every three months for each analyte used on each instrument. The linear range is the concentration above which results cannot be reported without dilution of the sample.

12.4.2 The LDR must be determined from a linear calibration prepared in the normal manner using the normal operating procedures described in Sections 10 and 11.

12.4.3 The LDR is determined by analyzing successively higher standard concentrations of the analytes of interest. A minimum of three standards are required for the initial and on-going studies, and one of the levels must be at the upper end of the range. The calculated concentrations must be within 10% of the stated concentrations.

12.4.4 The highest standard that meets this criterion defines the maximum concentration that can be reported for sample analysis without dilutions.

12.4.5 If the instrument is adjusted in any way that may affect the LDRs, new dynamic ranges must be determined. The LDR data must be documented and kept on file.

12.5 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.5.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.5.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.5.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.5.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.5.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.1 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and DV-HS-001P, *Waste Management Program*.

14.2 The following waste streams are produce when this method is carried out:

14.2.1 **Aqueous Acidic (Metals)** - Corrosive - Waste Stream J

14.2.2 **Expired reagents and standards –** Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition and all promulgated updates, EPA Office of Solid Waste, January 2005.

15.1.1 Method 6020A: *Inductively Coupled Plasma - Mass Spectrometry*, Revision 1, February 2007.

15.1.2 Method 6020B: Inductively Coupled Plasma - Mass Spectrometry, Revision 2, July 2014.

15.1.3 Method 3005A, Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy, Revision 1, July 1992.

15.1.4 Method 3020A, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA Spectroscopy, Revision 1, July 1992.

15.1.5 Method 3050B, Acid Digestion of Sediments, sludges and soils, Rev. 2, Dec. 1996.

15.2 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.2, 10/20/2010

15.3 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 5, July 2013.

16.0 Method Modifications:

Item	Method	Modification
1	EPA 6020A	Commercially available standards are purchased and verified at the laboratory rather than being prepared from the solid material. These verification records are kept on file with QA.
2	EPA 6020A	Milli-Q or Nanopure water is substituted when reagent water is called for. This water is tested to be free of contaminants by conductivity (18megOhm) and by the analysis of blanks.
3	EPA 6020A	Corrective action for a PDS failure will be limited to flagging the PDS indicating the failed analyte and the recovery rather than diluting and reanalyzing the sample.
4	EPA 6020A	Internal standard recoveries are based on the intensities of the internal standards in the most recent calibration blank rather than the intensities of the internal standards in the initial calibration standard.
5	EPA 6020A	Method 6020A states that the dilution test is applicable if the matrix sample is at least 50x the reporting limit. TestAmerica uses the tighter limit of 50x the MDL.
6	EPA 6020B	Method 6020B states to tune the instrument according to manufacturer's instructions. TestAmerica continues to use the tune requirements in Method 6020A in order to be able to run samples by either method under the same tune.

Item	Method	Modification
7	EPA 6020B	Method 6020B does not include the analysis of the ICS-AB. TestAmerica continues to analyze this QC sample due to various program requirements.
8	EPA 6020A/B	The tuning criteria listed in method 200.8 for mass resolution is used to satisfy the requirements of method 6020A/B

17.0 Attachments

Attachment 1: Standard Reporting Limits for Water and Soil
Attachment 2: Recommended Elemental Equations
Attachment 3: Isobaric Molecular-Ion Interferences Which Could Affect the Analytes
Attachment 4: Internal Standards and Corresponding Metals
Attachment 5: Interference Check Sample Components and Concentrations
Attachment 6: Suggested Mass Choices
Attachment 7: Tuning Solution and P/A Solution
Attachment 8: Suggested Tuning and Response Factor Criteria
Attachment 9: Summary of Quality Control Requirements
Attachment 10: Calibration, Calibration Verification, and Spike Concentrations
Attachment 11: Troubleshooting

18.0 Revision History

Revision 10, Dated 3 March 2020
- Updated Copyright Information

Revision 9, Dated 31 January 2020
- Annual Review
- Added 100 ml graduated cylinder to Section 6.2.3
- Updated standard information in Sections 7.2.3 - 7.2.5, 7.2.9.2 and 7.2.10
- Updated concentration levels in Attachments 1, 5, 7 and 10

Revision 8, Dated 31 October 2018
- Annual Review
- Updated copyright section

Revision 7, Dated 31 October 2017
- Annual Review
- Updated section 10.6.3 Mass Resolution criteria to method 200.8
- Added Section16 item 8 for Mass Resolution criteria

Revision 6, Dated 26 October 2016
- Updated sections 7.2.3.1.9, 7.2.4.1.8, 7.2.9.2.8 and 7.2.10 for how Sr and Li are prepared in the standards
- Updated Section 10.6.9 and Attachment 9 to reflect 6020B limits and use of in-house limits
- Updated Sections 10.6.10 and 10.6.11 for the state of Washington requirements

- Updated Section 15 for consistency with other SOPs to reflect reference for SW-846

Revision 5, Dated 28 September 2016

- Added Li and Sr as analytes to the SOP
- Revised Section 1.2 to more clearly identify elements addressed by this SOP
- Added prep method numbers to Sections 1.3 and 15
- Combined Footnotes 1 and 3 for table in Section 8.0 to eliminate redundancy
- Removed references to AFCEE throughout SOP; no longer utilize AFCEE protocols
- Expanded MS/MSD failure corrective action in Section 9.4 to reflect current policy
- Added note in Section 10.6.3 regarding Method 6020B requirements for tune
- Added LLICV acceptance criteria for Method 6020B, different from 6020A.
- Removed references to Instrument 24 (Agilent 7500) throughout
- Added requirements for IDLs by 6020B to Section 12.3
- Moved Section 12.5 to new Section 9.9
- Revised Sections 12.6 and 12.7 to reflect current practice
- Added Method exceptions 6 and 7 for Method 6020B where the laboratory performs a more stringent practice than required.

Revision 4, Dated 31 December 2015

- Added method 6020B
- Added 6020B limits to Section 9.7 and 9.8, SD and PDS
- Corrected references

Revision 3, Dated 30 April 2015

- Annual Review
- Language and formatting changes throughout
- Corrected tuning sample requirement from 4 replicates to five replicates
- Changed 12.4.2 to 12.5
- Added Section 2.1
- Added new Section 2.3 to describe reaction cell
- Added new Section 4.2.3
- Added batch definition
- Deleted Attachment 2
- Deleted Attachment 4
- Added new Attachment 1 standard reporting limits
- Section 4.3.2 enlarged expected IS intensities
- Integrated Sections 4.4 and 4.5 into 4.2
- Created new Section 4.3 Doubly charged ions
- Added new Section 6.2.3 volumetric flasks
- Section 7.2.1.4 corrected Mg addition to standard
- Section 7.2.2 replaced P/A section with same section from DV-MT-0025
- Added P/A section to Attachment 7
- Section 7.1.3 added reference to standards SOP
- Added all new standard prep information into Sections 7.2.3 – 7.2.9
- Section 9.3 changed spike level from midpoint of LR to midpoint of cal curve
- Added note to Section 9.4
- Changed timing of ICSA to beginning of analytical run and every 12 hours
- Changed concentration limits for SD in Section 9.7 to 50x MDL
- Added method modification 5 to address SD limit

- 10.6.7 added DoD 5.0 language to corrective action
- Removed Section 10.3
- 10.7.5 added DoD requirement to dilute above daily LR
- 11.5 Corrected RPD calculation
- 11.7 changed to dry weight correction
- 12.2.1 changed MDLV spike level to 2-4x MDL

Revision 2, dated 09 April 2014

- Annual review
- Updated Section 7.2 for standards to reference TALS for how to make
- Added Section 7.4.4 for 5%HNO3/5%HCL for Zirconium
- Added Section 10.4 for Maintenance
- Added Section 10.5 for Troubleshooting
- Updated Sections 9 and 10 to include requirements for DoD QSM 5.0
- Added reference to DoD QSM 5.0.

Revision 1, dated 15 July 2013

- Annual review
- Corrected formatting
- Added section 3.16
- Added reference to data review in section 10.7
- Added documentation information in section 11.8
- Added detail to note associated with section 14.2
- Updated reference in section 15.2
- Removed Attachment 13

Revision 0.3, dated 13 July 2012

- Revised standards preparation procedures in Section 7
- Added section 7.2.2
- Split acid diluent into two solutions depending upon instrument
- Updated standard mixes used to prepare standards; instrument specific mixes as needed
- Clarified requirements for preservation of soil samples for ICPMS only analysis, Section 8
- Revised list of common lab contaminants in method blank corrective action (Section 9.2)
- Added section 10.5.2: oxide/doubly charged performance check
- Updated Sections 9.2 and 10.5.7 to control method blanks and calibration blanks to ½ the RL
- Updated Sections 9.1, 10.1, 10.2 and 12.1 to reflect current practice.

Revision 0.2, dated 08 July 2011

- Added Section 4.4 on polyatomic interferences
- Added Instruments to Section 6.1
- Section 10.5.1 Added to condition cones with the ICSA solution
- Added Section 10.6.3 to reflect soil dilution practices
- Section 11.4 Corrected the RPD calculation
- Added section 11.1 referencing corporate SOP CA-Q-S-005 “Calibration Curves”
- Added section 12.2 “MDL Verification (MDLV)”
- Added Attachment 13 “ICP-MS Technical Data Review Checklist”

Attachment 1

Standard Reporting Limits for Water and Soil

Element Name	Element Symbol	Water (ug/L)	Soil (ug/Kg)
Aluminum	Al	50	5,000
Antimony	Sb	2.0	200
Arsenic	As	5.0	600
Barium	Ba	1.0	200
Beryllium	Be	1.0	100
Cadmium	Cd	1.0	100
Chromium	Cr	2.0	200
Cobalt	Co	1.0	100
Copper	Cu	2.0	250
Iron	Fe	50	5,000
Lead	Pb	1.0	150
Lithium	Li	50	5,000
Manganese	Mn	1.0	250
Molybdenum	Mo	2.0	200
Nickel	Ni	2.0	150
Selenium	Se	5.0	500
Silver	Ag	5.0	100
Strontium	Sr	10	100
Thallium	Tl	1.0	100
Thorium	Th	5.0	200
Tin	Sn	10	2,500
Tungsten	W	5.0	---
Uranium	U	1.0	100
Vanadium	V	5.0	500
Zinc	Zn	10	1,000
Zirconium	Zr	0.5	---

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Attachment 2

Recommended Elemental Equations

Element	Isobaric Correction	Mathematical Equation
Al	none	(1.0000)(27M)
Sb	none	(1.0000)(121M)
As	ArCl, Se	(1.0000)(75M) - (3.1278)(77M) + (1.0177)(78M)
Ba	none	(1.0000)(135M)
Be	none	(1.0000)(9M)
Cd	MoO, Sn	(1.0000)(114M) - (0.0268)(118M) - (1.0000)(135M)
Ca	none	(1.0000)(44M)
Cr	none	(1.0000)(52M)
Co	none	(1.0000)(59M)
Cu	none	(1.0000)(65M)
Fe	none	(1.0000)(57M)
Pb	none	(1.0000)(208M) + (1.0000)(207M) + (1.0000)(206M)
Mg	none	(1.0000)(25M)
Mn	none	(1.0000)(55M)
Ni	none	(1.0000)(60M)
K	none	(1.0000)(39M)
Se	Ar2	(1.0000)(78M) - (1.1869)(76M)
Ag	none	(1.0000)(107M)
Na	none	(1.0000)(23M)
Tl	none	(1.0000)(205M)
V	ClO, Cr	(1.0000)(51M) - (3.1081)(53M) + (0.3524)(52M)
Zn	none	(1.0000)(66M)
6Li	Li (natural)	(1.0000)(6M) - (0.0813)(7M)
Sc	none	(1.0000)(45M)
Y	none	(1.0000)(89M)
Rh	none	(1.0000)(103M)
In	Sn	(1.0000)(115M) - (0.0149)(118M)
Tb	none	(1.0000)(159M)
Ho	none	(1.0000)(165M)
Bi	none	(1.0000)(209M)

Where M = Total ion count rate at the specified mass.

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Attachment 3

Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{121}Sb	PdO		AgN			AgC	
^{123}Sb	AgO		AgN	SrCl	ZrS	CdC	
^{75}As	CoO	NiOH	NiN	ArCl	CaS	CuC	
^{138}Ba	SnO	SbOH					
^{137}Ba	SbO	SnOH		MoCl			
^{136}Ba	SnO	SnOH				SnC	
^{135}Ba	SnO	SnOH		MoCl			
^{134}Ba	SnO	SnOH	SnN	MoCl		SnC	
^{132}Ba	SnO, CdO	InOH	SnN	MoCl	MoS	SnC	
^{130}Ba	CdO	CdOH	SnN, CdN	MoCl	MoS	SnC	
^{9}Be							
^{114}Cd	MoO	MoOH	MoN	SeCl	SeS		
^{112}Cd	MoO, ZrO	MoOH	MoN	AsCl, SeCl	SeS	MoC	
^{111}Cd	MoO	MoOH	MoN	GeCl			
^{110}Cd	MoO, ZrO		MoN, ZrN	GeCl, AsCl	SeS	MoC	
^{113}Cd	MoO	MoOH		SeCl, AsCl			
^{116}Cd	MoO						
^{106}Cd	ZrO		MoN, ZrN		GeS	MoC, ZrC	
^{108}Cd	MoO, ZrO	ZrOH	MoN, ZrN	GeCl	SeS, GeS	MoC, ZrC	
^{52}Cr	ArO	ClOH				ArC	
^{53}Cr	ClO	ArOH	KN	NCl, OCl		KC	
^{50}Cr	SO		ArN		SO	ArC	Mo^{++}
^{54}Cr		ClOH	ArN, CaN			CaC	
^{59}Cr	CaO	CaOH	ScN	MgCl	AlS	TiC	Sn^{++}
^{63}Cu	TiO, PO_2	TiOH	TiN	SiCl, MgCl	PS	VC	ArNa
^{65}Cu	TiO	TiOH	VN	SiCl	SS, SO_2H	CrC	
^{208}Pb							
^{206}Pb							
^{207}Pb							
^{204}Pb							
^{55}Mn	KO	ArOH	KN		NaS	CaC	Cd^{++}

Attachment 3 (cont.)
Isobaric Molecular-Ion Interferences Which Could Affect the Analytes

Analyte	Interferences						
	Oxygen	Hydroxyl	Nitrogen	Chlorine	Sulfur	Carbon	Other
^{202}Hg	WO						
^{200}Hg	WO	WOH	WN				
^{199}Hg	WO	WOH					
^{201}Hg		WOH					
^{198}Hg	WO	TaOH	WN			WC	
^{204}Hg							
^{196}Hg			WN				
^{58}Ni	CaO	KOH	CaN	NaCl	MgS	TiC	Cd^{++}, Sn^{++}
^{60}Ni	CaO	CaOH	TiN	MgCl, NaCl	SiS	TiC	Sn^{++}
^{62}Ni	TiO	ScOH	TiN	AlCl, MgCl	SiS	TiC, CrC	Sn^{++}
^{61}Ni	SeO	CaOH	TiN	MgCl	SiS	TiC	
^{64}Ni	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{80}Se	ZnO	CuOH	ZnN	ScCl, CaCl	TiS	ZnC	
^{78}Se	NiO	NiOH	ZnN	CaCl, KCl	TiS	ZnC	
^{82}Se	ZnO	CuOH	ZnN	TiCl, ScCl	TiS, CrS		
^{76}Se	NiO	CoOH	NiN	KCl	CaS	ZnC	
^{77}Se	NiO	CuN	CuN	CaCl, ArCl	ScS	CuC	
^{74}Se	NiO	NiN	NiN	ClCl, KCl	CaS	NiC	
^{107}Ag	ZrO	ZrOH		GeCl	AsS	MoC	
^{109}Ag		MoOH	MoN	GeCl	SeS	MoC	
^{205}Tl							
^{203}Tl		WOH					
^{51}V	ClO	SOH	ClN	ClO, ClN	FS	KC	
^{50}V	SO		ArN			ArC	Mo
^{64}Zn	TiO	TiOH	TiN, CrN	SiCl, AlCl	SS	CrC	
^{66}Zn	TiO	TiOH	CrN	PCl, SiCl	SS	FeC	
^{68}Zn	CrO	VOH	FeN	PCl	ArS	FeC	Ba^{++}
^{67}Zn	VO	TiOH, Cr	CrN	SCl	ClS	MnC	Ba^{++}
^{70}Zn	FeO	CrOH	GeN	ClCl	ArS	NiC	

NOTE: The information provided in this table does not indicate that all of the described interferences need to be tested. However, the table can be consulted for informational purposes if unusual samples are encountered.

Attachment 4
Internal Standards and Corresponding Metals

IS	ICP-MS 077/078
^{6}Li	Be
Sc	Li, Na, Mg, Al, K, Ca
Ge	Sr, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Se
In	Mo, Ag, Cd, Sn, Sb, Ba
Ho	Tl, Pb, Th, U, W

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Attachment 5
Interference Check Sample Components and Concentrations

Interference Component	Solution A Concentration (mg/L)	Solution AB Concentration (mg/L)
Al	100.0	110.0
Ca	100.0	110.0
Fe	100.0	110.0
Mg	100.0	110.0
Na	100.0	110.0
P	100.0	100.0
K	100.0	110.0
S	100.0	100.0
C	200.0	200.0
Cl	1000.0	1000.0
Mo	2.0	2.1
Ti	2.0	2.1
As	0.0	0.1
Sb	0.0	0.1
Be	0.0	0.1
Ba	0.0	0.1
Cd	0.0	0.1
Cr	0.0	0.1
Co	0.0	0.1
Cu	0.0	0.1
Pb	0.0	0.1
Li	0.0	1.0
Mn	0.0	0.1
Ni	0.0	0.1
Nb	0.0	0.2
Pd	0.0	0.1
Pt	0.0	0.1
Se	0.0	0.1
Sr	0.0	0.1
Tl	0.0	0.1
Th	0.0	0.1
Sn	0.0	0.1
Ag	0.0	0.1
U	0.0	0.1
V	0.0	0.1
W	0.0	0.1
Zn	0.0	0.1

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Attachment 6
Suggested Mass Choices

Boldface masses indicate the masses which must have the most impact on data quality and the elemental equations used to collect the data. It is strongly recommended that elements other than those of interest be monitored to indicate other potential molecular interferences which could affect the data quality.

Mass	Element of Interest
"**27**"	Aluminum
121, "**123**"	Antimony
"75"	Arsenic
138, "137", 136, **135**, 134, 132, 130	Barium
"9"	Beryllium
114, 112, "111", 110, 113, 116, 106	Cadmium
42, 43, **44**, 46, 48	Calcium
"**52**", **53**, **50**, 54	Chromium
"**59**"	Cobalt
"**63**", **65**	Copper
56, **54**, **57**, 58	Iron
"**208**", "**207**", "**206**", 204	Lead
"7"	Lithium
24, **25**, **26**	Magnesium
"**55**"	Manganese
58, "**60**", 62, **61**, 64	Nickel
93	Niobium
105	Palladium
195	Platinum
39	Potassium
80, **78**, "**82**", **76**, **77**, 74	Selenium
"**107**", **109**	Silver
23	Sodium
"88"	Strontium
"**205**", 203	Thallium
232	Thorium
192	Tungsten
"**51**", **50**	Vanadium
64, "**66**", **68**, **67**, 70	Zinc
139	Lanthanum
118	Tin
238	Uranium
35, 37	Chlorine
98, 96, 92, **97**, 94, "95"	Molybdenum
72	Germanium (IS)
165	Holmium (IS)
115	Indium (IS)
6	Lithium (6+) (IS)
45	Scandium (IS)

Attachment 7: Tuning Solution and P/A Solution

A tuning solution containing elements representing all of the mass regions of interest must be analyzed. Below is a suggested solution covering a typical mass calibration range. Instrument manufacturer recommendations should be followed for tuning solutions.

The P/A solution is used to monitor the correlation between the Pulse and Analog parts of the electron multiplier. This solution is prepared at different concentrations depending on the current instrument conditions. The parent standard concentration is shown below.

Element	Tuning Concentration (µg/L)	P/A Concentration (mg/L)
Al		5
As		20
Ba	10	5
Be	40	20
Bi		5
Cd		20
Ce	10	
Co	10	5
Cr		5
Cu		5
Ge		10
In	10	5
Ir		5
^{6}Li		5
Li	10	
Lu		5
Mg	20	10
Mn		5
Mo		10
Na		5
Ni		10
Pb	10	10
Pd		10
Rh	10	
Ru		10
Sb		10
Sc		5
Sn		10
Sr		5
Tb		2.5
Th		50
Ti		50
Tl	10	50
U	10	50
V		50
Y	10	2.5
Zn		20

Attachment 8:

Suggested Tuning and Response Factor Criteria

Minimum Response from Tuning Solution:

Be	>1,000
Mg	>2,000
Rh	>20,000
Pb	>10,000
Li	>2,000
Co	>20,000
In	>1,000
Tl	>1,000

Suggested Mass Calibration:

Be	9.0122
Mg	23.98
Rh	102.91
Pb	207.98
Li	7.016
Co	58.9332
In	114.904
Tl	204.9744

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Attachment 9:

Summary of Quality Control Requirements

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
LLOQ (6020B only)	With initial setup, Quarterly and on an as needed basis	65 - 135% recovery or in-house limits	Terminate analysis; correct the problem; recalibrate.
LLICV (6020A only)	Beginning of every analytical run.	70 - 130% recovery. 6020A IS, 70-150% rec.	Terminate analysis; correct the problem; recalibrate.
ICV	Beginning of every analytical run.	90 - 110% recovery.	Terminate analysis; correct the problem; recalibrate.
ICB/CB	Immediately after each ICV	The result is < ½ RL.	Terminate analysis; correct the problem; recalibrate.
LLCCV (6020A only)	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	70 - 130% recovery. 6020A IS, 70-150% rec.	See Section 10.6.10. Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.
CCV	Beginning and end of run and every 10 samples OR every 2 hours, whichever is more frequent.	90 - 110% recovery.	Reanalyze twice in succession. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCV.

Attachment 9: Summary of Quality Control Requirements (Continued)

QC Parameter	Frequency*	Acceptance Criteria	Corrective Action
CCB	Immediately following each CCV.	The result must be < ½ RL. .	Reanalyze once. If acceptable, continue. If unacceptable, terminate analysis; correct the problem recalibrate the instrument, reverify calibration and rerun all samples since the last acceptable CCB.
ICSA	Beginning and every 12 hours.	Monitor for possible interferences.	See Section 9.5
ICSAB	Immediately following each ICSA.	Monitor for possible interferences.	See Section 9.5
Method Blank	One per lot of 20 field samples or fewer.	The result must be < ½ RL. Sample results greater than 10x the blank concentration or samples for which the contaminant is < RL, do not require redigestion or reanalysis.	Re-run once. If > ½ RL, redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Serial Dilution	One per batch of 20 field samples or fewer.	90 - 110% recovery	See Section 9.7 for additional requirements.
Post-Digestion Spike	One per batch of 20 field samples or fewer.	80-120% recovery	See Section 9.8.
Laboratory Control Sample	One per batch of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.3
Matrix Spike	One per lot of 20 field samples or fewer.	Must be within laboratory control limits	See Section 9.6 for additional requirements.

Attachment 10
Calibration, Calibration Verification, and Spike Concentrations

Element	Initial Calibration (µg/L)	ICV (µg/L)	CCV (µg/L)	LCS (µg/L)	MS/MSD (µg/L)	Post Digestion Spike (ug/L)	CCVL (ug/L)
Aluminum	2000	800	1000	400	400	20000	50
Antimony	100	40	50	40	40	200	2
Arsenic	100	40	50	40	40	200	5
Barium	100	40	50	40	40	200	1
Beryllium	100	40	50	40	40	200	1
Cadmium	100	40	50	40	40	200	1
Chromium	100	40	50	40	40	200	2
Cobalt	100	40	50	40	40	200	1
Copper	100	40	50	40	40	200	2
Iron	2000	800	1000	400	400	20000	50
Lead	100	40	50	40	40	200	1
Lithium	200	80	100	100	100	200	10
Manganese	100	40	50	40	40	200	1
Molybdenum	100	40	50	40	40	200	2
Nickel	100	40	50	40	40	200	1.5
Selenium	100	40	50	40	40	200	5
Silver	100	40	50	40	40	50	5
Strontium	200	80	100	40	40	200	1
Thallium	100	40	50	40	40	200	1
Thorium	100	40	50	40	40	--	2
Tin	100	40	50	40	40	200	10
Tungsten	100	40	50	40	40	200	5
Uranium	100	40	50	40	40	200	1
Vanadium	100	40	50	40	40	200	5
Zinc	100	40	50	40	40	200	10
Zirconium	100	40	50	40	40	--	--

This procedure has been developed for twenty elements. Additional elements may be included in the calibration solution at the above levels. Levels may be adjusted to meet specific regulatory or client programs.

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Attachment 11

ICP-MS Troubleshooting Guide

Problem	Possible Cause/ Solution
High Calibration Blanks	Inspect historical blank data to determine root cause Inspect, clean or replace torch Inspect, clean or replace pump tubing or sample tubing Inspect, clean or replace nebulizer Remake blank solution Recalibrate instrument
Instrument Drift	Make sure instrument has warmed properly Condition cones to aid stability Reslope to correct for changing cone conditions during run Stop run, clean cones and start over with a new calibration
Erratic Readings, High RSDs	Check nebulizer pressure Check sample flow around the pump, adjust tension on pump tubing to ensure smooth flow Check for clogs in the uptake tubing, nebulizer, or valve Clean or replace nebulizer
Low Sensitivity	Clean cones Adjust lens voltages Remove and clean lens, remove and clean or replace reaction cell
Bad Tune: Bad Mass Cal	Adjust lens voltages, remove and clean lens
Bad Tune: High Oxides	Inspect, clean, or replace torch, nebulizer, and spray chamber

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Accredited Laboratory

A2LA has accredited

TESTAMERICA DENVER

Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 12th day of August 2019.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2021

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

TESTAMERICA DENVER
4955 Yarrow Street
Arvada, CO 80002
Roxanne Sullivan Phone: 303-736-0100
www.testamericainc.com

ENVIRONMENTAL

Valid To: October 31, 2021 Certificate Number: 2907.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality Systems Manual for Environmental Laboratories), and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, ICP/MS, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, O_2), Oxygen Demand, Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Spectrophotometry (Automated), Titrimetry, Total Organic Carbon, Total Organic Halide

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Metals			
Aluminum	EPA 200.7	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Antimony	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Arsenic	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Barium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Beryllium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Boron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Cadmium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Calcium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Chromium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B

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Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Cobalt	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Copper	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Iron	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Lead	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Lithium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Magnesium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Manganese	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Mercury	EPA 245.1	EPA 7470A	EPA 7471A/7471B
Molybdenum	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nickel	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Potassium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Selenium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Silica	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silicon	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Silver	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Sodium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Strontium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Thallium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Thorium	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Tin	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Titanium	EPA 200.7	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Tungsten	----------	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Uranium	EPA 200.8	EPA 6020/6020A/6020B	EPA 6020/6020A/6020B
Vanadium	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Zinc	EPA 200.7/200.8	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B	EPA 6010B/6010C/6010D EPA 6020/6020A/6020B
Nutrients			
Nitrate (as N)	By calculation	EPA 300.0 EPA 9056/9056A By Calculation/Nitrate by Calc	EPA 9056/9056A By Calculation/Nitrate by Calc
Nitrate-nitrite (as N)	EPA 353.2	EPA 300.0 EPA 353.2 EPA 9056/9056A	EPA 9056/9056A
Nitrite (as N)	EPA 353.2 SM 4500-NO_2 B	EPA 300.0 EPA 353.2 EPA 9056/9056A SM 4500-NO_2 B	EPA 353.2 EPA 9056/9056A

Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Orthophosphate (as P)	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Total Phosphorus	EPA 365.1	EPA 6010B/6010C/6010D	EPA 6010B/6010C/6010D
Demands			
Total Organic Carbon	----------	EPA 9060/9060A	EPA 9060/9060A
Total Organic Halides	----------	EPA 9020B	----------
Wet Chemistry			
Alkalinity (Total Bicarbonate, Carbonate, and Hydroxide Alkalinty)	SM 2320B-1997	SM 2320B	SM 2320B
Ammonia	EPA 350.1	EPA 350.1	----------
Biological Oxygen Demand	SM 5210B	SM 5210B	----------
Bromide	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chloride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Chemical Oxygen Demand	EPA 410.4	EPA 410.4	----------
Conductivity	----------	EPA 9050/9050A	EPA 9050/9050A
Cyanide	----------	EPA 9012A/9012B	EPA 9012A/9012B
Ferrous iron	SM 3500Fe B, D	SM 3500Fe B, D	----------
Fluoride	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Flashpoint	----------	EPA 1010A	----------
Hexavalent chromium	----------	EPA 7196A	EPA 7196A
pH	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Oil and Grease (HEM and SGT-HEM)	----------	EPA 1664A/1664B	EPA 9071B
Percent moisture	----------	----------	ASTM D2216
Perchlorate	----------	EPA 6860	EPA 6860
Phenols	----------	EPA 9066	----------
Solids, total	----------	----------	SM 2540B
Solids, Total Suspended	SM 2540D	SM 2540D	SM 2540D
Solids, Total Dissolved	SM 2540C	SM 2540C	SM 2540C
Sulfate	----------	EPA 300.0 EPA 9056/9056A	EPA 9056/9056A
Sulfide, Total	SM 4500S2 D	EPA 9034/SM 4500S2 D	EPA 9034
Sulfide	----------	EPA 9030B	EPA 9030B
Total Kjeldahl Nitrogen	EPA 351.2	EPA 351.2	EPA 351.2
Purgeable Organics (Volatiles)			
Acetone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acetonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Acrolein	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Acrylonitrile	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Allyl Chloride	----------	EPA 8260B/8260C	EPA 8260B/8260C
Benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C/ AK101/OK DEQ GRO
Benzyl Chloride	----------	EPA 8260B/8260C/8260C	EPA 8260B/8260C/8260C

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Bromobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Bromodichloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromoform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Bromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Butadiene	----------	----------	----------
2-Butanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
n-Butyl alcohol	----------	EPA 8260B/8260C EPA 8015B/8015C	EPA 8260B/8260C EPA 8015B/8015C
tert-Butyl alcohol (2-Methyl-2-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
sec-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
tert-Butylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Carbon disulfide	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Carbon tetrachloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloro-1,3-butadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Chloroethyl vinyl ether	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroform	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1-Chlorohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Chloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Chloroprene	----------	EPA 8260B/8260C	EPA 8260B/8260C
4-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Chlorotoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Cyclohexanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dibromochloromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dibromo-3-chloropropane (DBCP)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
Dibromochloromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dibromomethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2 Dibromoethane (EDB)	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,2-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dichlorobenzene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,4-Dichloro-2-butene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,2-Dichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Dichlorodifluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dichlorofluoromethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2-Dichloropropane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2,2-Dichloropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1-Dichloropropene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
cis-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
trans-1,3-Dichloropropene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Diethyl ether	----------	EPA 8260B/8260C	EPA 8260B/8260C
Di-isopropylether	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Dioxane	EPA 624/624.1	EPA 8260B/8260C EPA 8260B/8260C SIM	EPA 8260B/8260C EPA 8260B/8260C SIM
Ethanol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl benzene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Ethyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Ethyl tert-butyl ether	----------	----------	----------
Gas Range Organics (GRO)	----------	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx	EPA 8015B/8015C/8015D/ AK101/OK DEQ GRO/NWTPH-Gx
Hexane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
2-Hexanone	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Hexachlorobutadiene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isobutyl alcohol (2-Methyl-1-propanol)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropyl alcohol	----------	EPA 8260B/8260C	EPA 8260B/8260C
Isopropylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,4-Isopropyltoluene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Iodomethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methacrylonitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl acetate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl cyclohexane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methylene chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Methyl ethyl ketone (MEK)	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl isobutyl ketone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl methacrylate	----------	EPA 8260B/8260C	EPA 8260B/8260C
Methyl tert-butyl ether (MtBE)	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
4-Methyl-2-pentanone (MIBK)	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Naphthalene	EPA 624/624.1	EPA 8260B/8260C OK DEQ GRO	EPA 8260B/8260C OK DEQ GRO
2-Nitropropane	----------	EPA 8260B/8260C	EPA 8260B/8260C
Pentachloroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
2-Pentanone	----------	EPA 8260B/8260C	EPA 8260B/8260C
Propionitrile	----------	EPA 8260B/8260C	EPA 8260B/8260C
n-Propylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Styrene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1,1,1,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2,2-Tetrachloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrachloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Tetrahydrofuran	----------	EPA 8260B/8260C	EPA 8260B/8260C
Toluene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
Total Petroleum Hydrocarbons (TPH)	EPA 1664A/1664B	EPA 1664A/1664B	----------
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,1,1-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,1,2-Trichloroethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichloroethene	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Trichlorofluoromethane	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trichlorobenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trichloropropane	EPA 624/624.1	EPA 8260B/8260C EPA 8011	EPA 8260B/8260C EPA 8011
1,1,2-Trichloro-1,2,2-trifluoroethane	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,3-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,2,4-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
1,3,5-Trimethylbenzene	----------	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl acetate	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Vinyl chloride	EPA 624/624.1	EPA 8260B/8260C	EPA 8260B/8260C
Xylenes, Total	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
1,2-Xylene (o-Xylene)	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/OK DEQ GRO
m+p-Xylene	EPA 624/624.1	EPA 8260B/8260C AK101/OK DEQ GRO	EPA 8260B/8260C AK101/ K DEQ GRO
Methane	----------	RSK-175	----------
Ethane	----------	RSK-175	----------
Ethylene (Ethene)	----------	RSK-175	----------
Acetylene	----------	RSK-175	----------
Acetylene ethane	----------	RSK-175	----------
Extractable Organics (Semivolatiles)			
Acenaphthene	EPA 625/625.1/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acenaphthylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Acetophenone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Acetylaminofluorene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Alachlor	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Aminobiphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Aniline	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Aramite	----------	EPA 8270C/8270D	EPA 8270C/8270D
Atrazine	----------	EPA 8270C/8270D	EPA 8270C/8270D
Azobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzaldehyde	----------	EPA 8270C 8270D	EPA 8270C/8270D
Benzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzoic acid	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Benzo(a)anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(b)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(k)fluoranthene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(ghi)perylene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzo(a)pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Benzyl alcohol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,1-Biphenyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethoxy) methane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroethyl) ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Chloroisopropyl) ether (2,2'Oxybis(1-chloropropane)	----------	EPA 8270C/8270D	EPA 8270C/8270D
bis (2-Ethylhexyl) phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Bromophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
butyl Benzyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-sec-butyl-4,6-Dinitrophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Caprolactam	----------	EPA 8270C/8270D	EPA 8270C/8270D
Carbazole	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chloroanilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Chlorobenzilate	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-chloro-3-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1-Chloronaphthalene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Chloronaphthalene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Chlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Chlorophenyl phenyl ether	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Chrysene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Cresols	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diallate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Dibenzo (a,h) anthracene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Dibenzofuran	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3,3'-Dichlorobenzidine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D

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Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
2,4-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethoate	----------	EPA 8270C/8270D	EPA 8270C/8270D
3,3-Dimethylbenzidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
p-Dimethylaminoazobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
7,12-Dimethylbenz(a)anthracene	----------	EPA 8270C/8270D	EPA 8270C/8270D
alpha-, alpha-Dimethylphenethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dimethylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dimethyl phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-butyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
di-n-octyl Phthalate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4,6-Dinitro-2-methylphenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,6-Dinitrotoluene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Dinoseb	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,4-Dioxane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Diphenylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2-Diphenylhydrazine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Disulfoton	----------	EPA 8270C/8270D	EPA 8270C/8270D
Diesel Range Organics (DRO)	----------	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx	EPA 8015B/8015C/8015D AK102/8015D/OK DEQ DRO/NWTPH-Dx
Ethyl Methanesulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D
Famphur	----------	EPA 8270C/8270D	EPA 8270C/8270D
Fluoroanthene	EPA 625/625.1	EPA 8270C/8270D EPA8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Fluorene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Hexachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorobutadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorocyclopentadiene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloroethane	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Hexachlorophene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Hexachloropropene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Indeno (1,2,3-cd) pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270C SIM/8270D SIM	EPA 8270C/8270D EPA 8270C SIM/8270D SIM
Isodrin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Isophorone	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Isosafrole	----------	EPA 8270C/8270D	EPA 8270 C/8270D
Methapyrilene	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Methylcholanthrene	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-methyl-4,6-Dinitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
methyl Methane sulfonate	----------	EPA 8270C/8270D	EPA 8270C/8270D

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
1-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylnaphthalene	----------	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
2-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
3+4-Methylphenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Naphthalene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
1,4-Naphthoquinone	----------	EPA 8270C/8270D	EPA 8270C/8270D
1-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Naphthylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
3-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitroaniline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Nitrobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
4-Nitrophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Nitroquinoline-1-oxide	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodimethylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-butylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodi-n-propylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosodiphenylamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomethylethylamine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosomorpholine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopiperidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
N-Nitrosopyrrolidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
5-nitro-o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,2-oxybis(1-chloropropane)	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, methyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Parathion, ethyl	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloroethane	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachloronitobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pentachlorophenol	EPA 625/625.1	EPA 8270C/8270D EPA 8321A/8321B	EPA 8270C/8270D EPA 8321A/8321B
Phenacetin	----------	EPA 8270C/8270D	EPA 8270C/8270D
Phenanthrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Phenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
p-Phenylene Diamine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Phorate	----------	EPA 8270C/8270D	EPA 8270C/8270D
2-Picoline	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pronamide	----------	EPA 8270C/8270D	EPA 8270C/8270D
Pyrene	EPA 625/625.1	EPA 8270C/8270D EPA 8270D SIM	EPA 8270C/8270D EPA 8270D SIM
Pyridine	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Safrole	----------	EPA 8270C/8270D	EPA 8270C/8270D

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Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Sulfotepp	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4,5-Tetrachlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,3,4,6-Tetrachlorophenol	----------	EPA 8270C/8270D	EPA 8270C/8270D
Thionazin	----------	EPA 8270C/8270D	EPA 8270C/8270D
o-Toluidine	----------	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Tribromophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
Tributyl phosphate	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
1,2,4-Trichlorobenzene	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,5-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
2,4,6-Trichlorophenol	EPA 625/625.1	EPA 8270C/8270D	EPA 8270C/8270D
o,o,o-triethyl Phosphorothioate	----------	EPA 8270C/8270D	EPA 8270C/8270D
1,3,5-Trinitrobenzene	----------	EPA 8270C/8270D	EPA 8270C/8270D
Motor Oil (Residual Range Organics)	----------	EPA 8015B/8015C/8015D AK103/OK DEQ RRO	EPA 8015B/ 8015C/8015D AK103/ OK DEQ RRO
Pesticides/Herbicides/PCBs			
Aldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Atrazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos ethyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
Azinophos methyl	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
beta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
delta-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-BHC	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Bolstar	----------	EPA 8141A/8141B	EPA 8141A/8141B
alpha-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
gamma-Chlordane	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chlordane (technical)	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Chloropyrifos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Coumaphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4-D	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dalapon	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
2,4-DB	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
4,4'-DDD	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDE	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
4,4'-DDT	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Demeton-O	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton-S	----------	EPA 8141A/8141B	EPA 8141A/8141B
Demeton, total	----------	EPA 8141A/8141B	EPA 8141A/8141B
Diazinon	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dicamba	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Dichlorovos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dichloroprop	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A

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Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Dieldrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Dimethoate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Dinoseb	----------	EPA 8151A EPA 8321A	EPA 8321A
Disulfoton	----------	EPA 8141A/8141B	EPA 8141A/8141B
Endosulfan I	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endosulfan II	EPA 608/608.3	EPA 8081A /8081B	EPA 8081A/8081B
Endonsulfan sulfate	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin aldehyde	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Endrin ketone	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
EPN	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethoprop	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ethyl Parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Famphur	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fensulfothion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Fenthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Heptachlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Heptachlor epoxide	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Hexachlorobenzene	----------	EPA 8081A/8081B	EPA 8081A/8081B
Malathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
MCPA	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
MCPP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA8321A
Merphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Methoxychlor	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Methyl parathion	----------	EPA 8141A/8141B	EPA 8141A/8141B
Mevinphos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Naled	----------	EPA 8141A/8141B	EPA 8141A/8141B
PCB-1016 (Arochlor)	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1221	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1232	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1242	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1248	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1254	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1260	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1262	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
PCB-1268	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Total PCBs	EPA 608/608.3	EPA 8082/8082A	EPA 8082/8082A
Pentachlorophenol	----------	EPA 8151A	EPA 8151A
Phorate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Phosmet	----------	EPA 8141A/8141B	EPA 8141A/8141B
Picrolam	----------	EPA 8151A	EPA 8151A
Propazine	----------	EPA 8141A/8141B	EPA 8141A/8141B
Ronnel	----------	EPA 8141A/8141B	EPA 8141A/8141B
Simazine	----------	EPA 8141A/8141B	EPA 8141A/8141B

Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
Stirophos	----------	EPA 8141A/8141B	EPA 8141A/8141B
Sulfotepp	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-T	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Thionazin	----------	EPA 8141A/8141B	EPA 8141A/8141B
Tokuthion	----------	EPA 8141A/8141B	EPA 8141A/8141B
2,4,5-TP	----------	EPA 8151A EPA 8321A	EPA 8151A EPA 8321A
Toxaphene	EPA 608/608.3	EPA 8081A/8081B	EPA 8081A/8081B
Trichloronate	----------	EPA 8141A/8141B	EPA 8141A/8141B
o,o,o-Triethylphos Phorothioate	----------	EPA 8141A/8141B	EPA 8141A/8141B
Explosives			
1,3,5-Trinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8330B	EPA 8330B
2,4-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-amino-4,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-amino-2,6-Dinitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitrobenzene	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Nitroglycerin	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Picric acid	----------	EPA 8330A/8330B	EPA 8330A/8330B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8330A/8330B EPA 8321A/8321B	EPA 8330A/8330B EPA 8321A/8321B
DNX	----------	EPA 8330A/8330B	EPA 8330A/8330B

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Parameter/Analyte	Non-Potable (Water)	Solid Hazardous Waste (Water)	Solid Hazardous Waste (Solid)
MNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
TNX	----------	EPA 8330A/8330B	EPA 8330A/8330B
Explosives LC/MS/MS			
1,3,5-Trinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
1,3-Dinitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4,6-Trinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3,5-Dinitroaniline	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Diamino-4-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,4-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Diamino-6-nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2,6-Dinitroltoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Amino-4,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
2-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
3-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Amino-2,6-Dinitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
4-Nitrotoluene	----------	EPA 8321A/8321B	EPA 8321A/8321B
DNX (hexahydro-1,3-dinitroso-5-nitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
MNX (hexahydro-1-nitroso-3,5-dinitro-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitrobenzene	----------	EPA 8321A/8321B	EPA 8321A/8321B
Nitroglycerin	----------	EPA 8321A/8321B	EPA 8321A/8321B
HMX (octahydro-1,3,5,7-tetrabitro-1,3,5,7-Tetrazocine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Pentaerythritoltetranitrate (PETN)	----------	EPA 8321A/8321B	EPA 8321A/8321B
RDX (hexahydro-1,3,5-trinitro-1,3,5-Triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tetryl (methyl 2,4,6-Trinitrophenylnitramine	----------	EPA 8321A/8321B	EPA 8321A/8321B
TNX (hexahydro-1,3,5-trinitroso-1,3,5-triazine)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Tris(o-cresyl)phosphate	----------	EPA 8321A/8321B	EPA 8321A/8321B
Chemical Warfare Agents			
Thiodiglycol (2,2'-Thiodiethanol)	----------	EPA 8321A/8321B	EPA 8321A/8321B
Hazardous Waste Characteristics			
Conductivity	SM 2510B	EPA 9050A	EPA 9050A
Corrosivity	SM 4500 H+B	EPA 9040B/9040C	EPA 9045C/9045D
Paint filter liquids test	----------	EPA 9095A	EPA 9095A
Synthetic Precipitation Leaching Procedure (SPLP)	----------	EPA 1312	EPA 1312
Toxicity Characteristic Leaching Procedure	----------	EPA 1311	EPA 1311
Turbidity	EPA 180.1	----------	----------
Organic Prep Methods			
Separatory funnel liquid-liquid extraction	----------	EPA 3510C	----------

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Parameter/Analyte	**Non-Potable (Water)**	**Solid Hazardous Waste (Water)**	**Solid Hazardous Waste (Solid)**
Continuous liquid-liquid extraction	----------	EPA 3520C	----------
Soxhlet extraction	----------	----------	EPA 3540C
Microwave extraction	----------	----------	EPA 3546
Ultrasonic extraction	----------	----------	EPA 3550B/3550C
Waste dilution	----------	EPA 3580A	EPA 3580A
Solid phase extraction	----------	EPA 3535A	----------
Volatiles purge and trap	----------	EPA 5030B	EPA 5030A EPA 5035/5035A
Organic Cleanup Procedures			
Florisil cleanup	----------	EPA 3620B	EPA 3620B
Florisil cleanup	----------	EPA 3620C	EPA 3620C
Sulfur cleanup	----------	EPA 3660A	EPA 3660A
Sulfuric acid/Permanganate cleanup	----------	EPA 3665A	EPA 3665A
Metals Digestion			
Acid digestion total recoverable or dissolved metals	----------	EPA 3005A	----------
Acid digestion for total metals	----------	EPA 3010A	----------
Acid digestion for total metals	----------	EPA 3020A	----------
Acid digestion of sediments, sludges and soils	----------	----------	EPA 3050B

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, and for the test methods applicable to the Wyoming Storage Tank Remediation Laboratory Accreditation Program), accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

WYOMING STORAGE TANK PROGRAM

Parameter/Analyte	**Method(s)**
Metals	
Cadmium	EPA 6010C/6010D
Chromium	EPA 6010C/6010D
Lead	EPA 6010C/6010D
Wet Chemistry	
Hexavalent chromium	EPA 7196A
Pureable Organics (Volatiles)	
tert-Amyl Methyl Ether	EPA 8260B/8260C
Benzene	EPA 8260B/8260C
tert-Butyl alcohol (2-Methyl-2-propanol)	EPA 8260B/8260C
1,2-Dichloroethane	EPA 8260B/8260C
Di-isopropylether	EPA 8260B/8260C
Ethyl benzene	EPA 8260B/8260C
Ethyl tert-butyl ether	EPA 8260B/8260C
Gas Range Organics (GRO)	EPA 8015B/8015C/8015D

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Parameter/Analyte	**Method(s)**
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C
Naphthalene	EPA 8260B/8260C
Toluene	EPA 8260B/8260C
Xylenes, total	EPA 8260B/8260C
1,2-Xylene	EPA 8260B/8260C
M+P-Xylene	EPA 8260B/8260C
Extractable Organics (Semivolatiles)	
Diesel Range Organics (DRO)	EPA 8015B/8015C/8015D (WY: C10-C32)
Organic Prep Methods	
Volatiles purge and trap	EPA 5030B (water) /5030A (solids)

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Accredited Laboratory

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Arvada, CO

for technical competence in the field of

Environmental Testing

In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2017, the 2009 TNI Environmental Testing Laboratory Standard, the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP), and the requirements of the Department of Energy Consolidated Audit Program (DOECAP) as detailed in version 5.3 of the DoD/DOE Quality System Manual for Environmental Laboratories (QSM), accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 12th day of August 2019.

Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2907.01
Valid to October 31, 2021

For the tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

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Title: Mercury in Solids by Cold Vapor Atomic Absorption

Approvals (Signature/Date):

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1.0 Scope and Application

1.1 This procedure describes the preparation and analysis of mercury (Hg, CAS# 7439-97-6) by Cold Vapor Atomic Absorption Spectroscopy (CVAA) using SW-846 Method 7471A or 7471B.

1.2 Method 7471A and 7471B are applicable to the preparation and analysis of mercury in soils, sediments, bottom deposits, and sludge-type materials. All matrices require sample preparation prior to analysis. This is not an appropriate procedure for the digestion of tissues or other organic matrices, which require the use of EPA 245.6 instead.

1.3 If sample preparation utilizing the Incremental Sampling Method is required, see SOP DV-OP-0013 for the procedure required prior to acid digestion for metals incorporating this procedure.

1.4 The routine reporting limit for mercury in solid matrices is 17 µg/kg.

2.0 Summary of Method

A representative portion of the sample is digested in aqua regia in the first digestion cycle and potassium permanganate in the second cycle. Mercury is reduced to its elemental state with stannous chloride and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorption of light at 253.7 nm is calibrated as a function of mercury concentration.

3.0 Definitions

3.1 Total Mercury: Inorganic forms of mercury are effectively dissolved by the acids used in the digestion. The potassium permanganate reagent breaks down organo-mercury compounds to inorganic forms that are detected by this procedure.

3.2 Aqua Regia: A 3:1 mixture of hydrochloric and nitric acids. This mixture is effective at dissolving metals in the solid form.

3.3 General Analytical Terms: Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and Policy DV-QA-003P, *Quality Control Program*, for definitions of general analytical and QA/QC terms.

4.0 Interferences

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1 Potassium permanganate "suitable for mercury determination" is specified because of the potential for mercury contamination in the reagent. In addition, potassium permanganate crystals will absorb mercury vapors from the air. Reagent bottles must be kept tightly closed to avoid contamination.

4.2 Potassium permanganate, in addition to breaking down organic compounds, also

eliminates possible interferences from sulfide. Concentrations as high as 20 ppm of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from reagent water.

4.3 Copper has also been reported to interfere; however, copper concentrations as high as 10 ppm had no effect on the recovery of mercury from spiked samples.

4.4 Chlorides can cause a positive interference. Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation at 253.7 nm. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by adding excess hydroxylamine reagent (25 mL) before stannous chloride is added. Both inorganic and organic mercury spikes have been quantitatively recovered from seawater using this technique.

NOTE: Sufficient addition of permanganate is apparent when the purple color persists at least 15 minutes. Some samples may require dilution prior to digestion due to extremely high concentrations of chloride.

4.5 Interference from certain volatile organic materials that absorb at the wavelength used for the method may also occur. If suspected, a preliminary run without stannous chloride can determine if this type of interference is present. While the possibility of absorption from certain organic substances present in the sample does exist, this problem is not routinely encountered. This is mentioned only to caution the analyst of the possibility. If this condition is found to exist, the mercury concentration in the sample can be determined by subtracting the result of the sample run without the reducing reagent (stannous chloride) from that obtained with the reducing reagent.

4.6 Samples containing high concentrations of oxidizable organic materials, as evidenced by high COD levels, may not be completely oxidized by this procedure. When this occurs, the recovery of mercury will be low. The problem can be lessened by reducing the volume of original sample used.

4.7 The most common interference is laboratory contamination which may arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe,

nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Eye protection that satisfies ANSI Z87.1, laboratory coat, and nitrile or latex gloves must be worn while handling samples, standards, solvents, and reagents. Disposable gloves that have been contaminated must be removed and discarded; non-disposable gloves must be cleaned immediately. A disposable face shield should be used when making up aqua regia.

5.3.2 Potassium permanganate is a strong oxidizing agent. It is incompatible and must be stored separately from hydroxylamine hydrochloride and stannous chloride, the reducing agents used in this procedure, and from acids.

5.4 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury Nitrate Solutions	Corrosive Poison	0.1 mg/m^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydroxylamine Hydrochloride	Corrosive Poison	No OSHA PEL listed for this compound	Direct contact with skin or eyes causes irritation. May cause skin sensitization, an allergic reaction. Inhalation or ingestion may cause methemoglobinemia and resulting cyanosis (bluish discoloration of skin due to deficient oxygenation of the blood), and labored breathing.
Potassium Permanganate	Oxidizer	5 mg/m^3 for Mn compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions. 2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

6.1 Instrumentation

6.1.1 Digestion Block, with adjustable heating, capable of maintaining a sample temperature of 90 – 95 °C.

6.1.2 Mercury Autoanalyzers:

CETAC Mercury Analyzer with Autosampler and Auto-Diluter

6.2 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

6.3 Supplies

6.3.1 Disposable digestion tubes with caps from Environmental Express, volume accuracy verified to ASTM Class A standards at all gradations.

6.3.2 Disposable glass autosampler tubes, 16 mm x 100 mm

6.3.3 Argon, 99.999% purity

6.3.4 Calibrated mechanical pipettes (see SOP DV-QA-0008 for details on calibrating mechanical pipettes).

6.3.5 Class A volumetric flasks.

6.3.6 Thermometer, non-mercury column, accurate to ± 1 °C at 95 °C (see SOP DV-QA-0001 for calibration details).

6.3.7 Glass beads, < 1 mm diameter, acid washed.

7.0 Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Primary and secondary standards used for data production are recorded in the Reagent Module of the TestAmerica LIMS (TALS).

7.1 **Reagent water:** Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

7.2 **Nitric acid (HNO_3):** Concentrated, trace metal grade or better.

7.3 **Hydrochloric acid (HCl):** Concentrated, trace metal grade or better.

7.4 **Aqua Regia:** Add 75 mL concentrated HCL and 25 mL concentrated HNO3 to a 100 mL container. Aqua Regia will be prepared immediately before use.

7.5 **Calibration Blank, Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Method Blank (MB), 1% HNO_3:**

7.5.1 Add 0.5 L of concentrated HNO_3 to a 50 L carboy partially filled with reagent water.

7.5.2 Dilute to 50 L with reagent water.

7.5.3 Record the acid lot number and other required information in the Blank Reagent Logbook stored in the metals prep area.

7.6 **Stannous chloride solution ($SnCl_2$), Hg grade, 10% (w/v) per manufacturer's instructions:**

7.6.1 Place approximately 100 mL of reagent water into a 2 L volumetric flask.

7.6.2 Slowly add 200 mL of concentrated HCl to the flask and swirl to mix.

7.6.3 Add 200 grams of $SnCl_2$ to the flask.

7.6.4 Mix the contents of the flask until the reagent is completely dissolved.

7.6.5 Bring solution to a final volume of 2 L with reagent water.

7.7 Sodium chloride-hydroxylamine hydrochloride solution (Hg grade):

Add 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride (Hg grade) to every 100 mL of reagent water.

NOTE: Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.

7.8 Potassium permanganate, 5% solution (w/v):

Dissolve 5 g of potassium permanganate (reagent grade, "suitable for mercury determination") for every 100 mL of reagent water.

7.9 Purchased Mercury Stock Solutions

7.9.1 Second source initial calibration verification (ICV) stock solution 100 mg/L (Hg ICV Stock).

7.9.2 Primary Mercury Calibration Standard Solution, 1,000 mg/L (Hg Ultra Prim).

7.10 Monthly Calibration Working Standard Solution, 10 mg/L (Hg Mnth Spike)

7.10.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL Class A volumetric flask.

7.10.2 Pipet 1.0 mL of the 1,000 mg/L primary mercury calibration standard solution (Section 7.9.2) into the flask.

7.10.3 Dilute to the mark on the flask with 1% HNO_3.

7.10.4 Stopper the flask and shake to mix.

7.10.5 Transfer the solution to a 125 mL Nalgene bottle.

7.10.6 Document the preparation of the solution in the Reagent Module in TALS.

7.10.7 Prepare this solution fresh monthly or more often if necessary.

7.11 Daily Calibration Working Spike, 100 µg/L (Hg Daily Spk)

7.11.1 Add approximately 90 mL of 1% HNO_3 to a 100 mL volumetric flask.

7.11.2 Add 1.0 mL of the 10 mg/L Calibration Working Standard (see Section 7.10).

7.11.3 Bring the solution to a final volume of 100.0 mL.

7.11.4 Stopper and mix thoroughly.

7.11.5 Document the preparation in the Reagent Module in TALS.

7.11.6 Prepare this solution each day prior to calibration.

7.12 Initial Calibration (ICAL) Standards

The initial calibration standards are prepared directly in the digestion tubes as follow:

ICAL	Daily Calibration Working Spike (mL)	1% HNO_3 (mL)	Final Conc. (µg/L)
Blank	0.0	5.0	0.0
Std 1	0.10	4.9	0.20
Std 2	0.25	4.75	0.50
Std 3	0.50	4.5	1.0
Std 4	1.0	4.0	2.0
Std 5	2.5	2.5	5.0
Std 6	5.0	0.0	10.

7.13 Second-Source Initial Calibration Verification Intermediate Standard, 400 µg/L (Hg Biwk ICV)

Add 40 µL of the 1000 mg/L ICV Standard to a 100 mL volumetric flask partially filled with 1% HNO_3 and dilute to the mark. Record this information in the Reagent Module in TALS.

7.14 Second-Source Initial Calibration Verification (ICV) Daily Working Standard, 4.00 µg/L (Hg Soil ICV)

Add 0.5 mL of the 400 µg/L ICV Intermediate Standard (see Section 7.13) to a soil digestion tube and add 4.5 mL of 1% HNO_3. Record this information in the Reagent Module in TALS.

7.15 Continuing Calibration Verification (CCV) Standards, 5 µg/L (Hg Soil CCV)

7.15.1 The CCVs are prepared exactly as the 5.0 µg/L ICAL standard shown above (see Section 7.12).

7.15.2 Prepare sufficient volume of the standard for analysis of a CCV after every 10 samples.

7.16 Laboratory Control Sample (LCS), 333 µg/kg

7.16.1 The LCS is prepared in an empty digestion tube or 0.6 g of glass beads (< 1 mm) are used if required.

7.16.2 Add 2.0 mL of the 100 µg/L Daily Calibration Working Spike (see Section 7.11) to a digestion tube. See Section 9.4 for additional detail.

7.16.3 This is equivalent to 4.0 µg/L, which is the concentration that appears on the raw data printout from the instruments.

7.17 Matrix Spike and Matrix Spike Duplicate (MS/MSD), 333 µg/kg

MS/MSD pairs are spiked in the same manner as the LCS (see section 7.16) and prepared in the same manner as the samples, using 0.6 g of sample.

7.18 Reporting Limit (CRA) Check Standard, 0.2 µg/L (Hg Soil RL)

7.18.1 Add 0.1 mL of 100 µg/L Daily Calibration Working Spike (see section 7.11) and 4.9 mL of reagent water to a digestion tube.

7.18.2 This is equivalent to a 0.2 µg/L ICAL standard, which is the concentration that appears on the raw data printout from the instruments.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	10 grams	Cool, ≤ 6 °C	28 Days	N/A

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the TestAmerica LIMS (TALS) Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver Policy DV-QA-003P, *Quality Control Program.*

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver Policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for the DoD/DOE QSM (5.0 – 5.3) unless otherwise stated. Any deviation or exceptions

from QSM requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in TALS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Preparation Batch

A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, an LCS, and a matrix spike/matrix spike duplicate pair (MS/MSD). As discussed in the following sections, special program or project requirements can include additional requirements. Always refer to special project instructions for details before proceeding with the analysis.

9.3 Method Blank (MB)

The MB consists of an empty vessel (or with glass beads for DoD/DOE projects) containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. At least one method blank (MB) must be processed with each preparation batch.

Acceptance Criteria: The result for the method blank must be less than the project-specific data quality objectives. In the absence of project-specific data quality objectives, the blank must be less than ½ RL or less than 10% of the mercury concentration found in the associated samples, whichever is higher.

Corrective Action: All samples associated with an unacceptable method blank must be re-prepared and reanalyzed. If mercury was not detected in the samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative.

NOTE: DOD QSM 5 does not allow this exception. Results may not be reported without a valid

method blank unless sample cannot be re-prepared or re-analyzed.

9.4 Laboratory Control Sample (LCS), 333 µg/kg

The preparation of the LCS is described in Section 7.16. At least one LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. DoD requires when there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Maximum control limits for LCS recoveries are 80 - 120%. In-house control limits based on three standard deviations of the mean of historical results are used as long as they are at least as tight as 80-120% (see Policy DV-QA-003P for further details on establishing control limits).

NOTE: DOD QSM 5 Solid matrix LCS Limits are 80 - 124%.

Corrective Action: If LCS recoveries are outside established control limits, the system is out of control and corrective action must occur. If recoveries are above control limits and mercury is not detected in samples, the data may be reported with qualifiers (check project requirements to be sure this is allowed) and it must be addressed in the project narrative. In other circumstances, the entire batch must be re-prepared and reanalyzed.

NOTE: Reporting sample results with failing LCS recoveries is not acceptable for DOD QSM 5 unless samples cannot be re-prepared or reanalyzed.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD), 333 µg/kg

One MS/MSD pair must be processed for each preparation batch. Some programs may require the use of sample duplicates in place of or in addition to MS/MSDs. In addition, some programs will allow spikes to be reported only for project-related samples. Samples identified as field blanks cannot be used for MS/MSD analysis. DoD requires the MS/MSD to be assigned by the client. When there is no assigned MS/MSD or there is not enough sample volume provided an LCSD must be prepared.

Acceptance Criteria: Control limits are statistically determined based on three standard deviations of the mean of the laboratory's historical data. The MS/MSD recovery must fall within 75-125%; the relative percent difference (RPD) between the MS and MSD cannot exceed 20%.

NOTE: DOD QSM 5 Solid matrix MS/MSD Limits are 80-124%.

Corrective Action: The information obtained from MS data are sample/matrix specific and are not normally used to determine the validity of the entire batch. If the MS and/or MSD recovery falls outside of the established control limits, the bracketing CCV and batch LCS recoveries must be within control limits in order to accept results for the associated samples. The following corrective actions are required for MS/MSD recovery failures to rule out lab error:

- Check calculation and instrument performance;
- Verify, if possible, that the MS and MSD were spiked correctly (e.g., very low or very high recoveries);
- Consider objective evidence of matrix interference (e.g., heterogeneous sample, interfering peaks seen on chromatograms, or interference demonstrated by prior analyses);
- Flag the data for any results outside of acceptance limits and note it on the final report.
- For any single RPD failure, check calculations; verify, if possible, that the MS and MSD were spiked correctly; check instrument performance; consider objective evidence of matrix interference or sample inhomogeneity; and flag the data.
- If both the parent sample and associated matrix spike results are over range the parent and the spikes shall be diluted by the same amount and the results from the reanalysis reported for both. If the analyte concentration in the parent sample is greater than four times the concentration of spike added, then spike recovery results are not compared to control limits, and the recovery is either reported as “NC” (not calculated) or with a qualifier flag to indicate that the spike was less than four times the analyte concentration in the sample. If the dilution will cause the spike to be less than two times the reporting limit, the MS/MSD do not need to be diluted and the recovery reported as “NC” (not calculated).
- For MS/MSD that serve as batch QC, if the parent sample result is within the linear range and the MS/MSD results are above the linear range, the results are reported with the MS/MSD result being flagged as an over-range measurement (e.g., the E-flag qualifier).
- If the MS/MSD are client requested, the parent sample result is within linear range and the MS/MSD results

are above the linear range, the sample and spike should be diluted, keeping in mind that we need to assess whether or not the dilution will best serve the client's needs. Consult with the PM as needed. Both the parent sample and MS/MSD samples must have the same dilution factor. Some EDDs do not accept data that are at different dilution factors.

- If the native analyte concentration in the MS/MSD sample exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated) and the appropriate qualifier flags are added.

NOTE 1: Some client programs require reanalysis to confirm matrix interferences. Check special project requirements for this corrective action.

NOTE 2: See Denver Policy Memorandum P16-001 and Corporate Policy Memorandum CA-Q-QM-013 for more detail.

9.6 Method of Standard Addition (MSA)

The method of standard additions is an option for the analysis of samples shown to have significant matrix effects, e.g., unacceptably low MS/MSD recoveries or under certain conditions for TCLP analysis (see Attachment 2 for details).

NOTE: DOD QSM 5: Performed only when required by the project and the SD or PDS fails. Must be documented with an NCM and included in the case narrative.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Sample Preparation

All equipment IDs for any support equipment (pipettes, thermometers, etc.) must be recorded in the batch record.

10.3.1 All calibration and calibration verification standards (ICV, ICB, CCV, CCB), as well as the field samples, are processed through the digestion procedure. Prepare digestion tubes containing volumes of standards required for each tube as listed in Section 7.

10.3.2 Weigh 0.5 – 0.6 g of each well-homogenized sample into a sample digestion tube. See SOP DV-QA-0023 for additional information on subsampling.

10.3.3 If preparing Incremental Samples a 3 g sample aliquot is required. This aliquot will be prepared by utilizing the procedure described in *Incremental Sampling Methodology for Soils and Sediments* (DV-OP-0013). Divide the 3 g aliquot into five 0.6 g samples. Digest the five individual aliquots and combine them back into one after adding the sodium chloride-hydroxylamine hydrochloride reagent. All batch QC samples must also be processed in this fashion.

10.3.4 Prepare a MB, LCS, MS, and MSD for each batch. The MB is either an empty digestion tube or is prepared by placing 0.6 g of glass beads in a digestion tube, depending on client requirements. The LCS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube. The MS is prepared by adding 2.0 mL of the 100 µg/L Daily Calibration Working Spike to a digestion tube containing a second aliquot of the chosen matrix sample. The MSD is prepared in the same manner as the MS using a third aliquot of the chosen sample.

NOTE: The spike must be added after the sample aliquot but before the addition of reagents.

10.3.5 Add 5.0 mL of reagent water to all un-spiked field samples and the method blanks. Add 3.0 mL of reagent water to the LCS, MS and MSD.

10.3.6 Add 5.0 mL of Aqua Regia to each tube.

10.3.7 Heat for 2 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS.

10.3.8 Allow the samples and standards to cool at room temperature.

10.3.9 Add 19 mL of reagent water.

10.3.10 Add 15 mL of 5% potassium permanganate solution. A purple color must persist for at least 15 minutes. If the color does not persist, the sample must be re-prepared using a smaller sample aliquot.

NOTE: It is important that equal volumes of the potassium

permanganate solution are added to all solutions in the batch. Unequal volumes used with the automated method will result in dilution errors.

10.3.11 Cap the samples and standards and heat for 30 minutes at 95 ± 3 °C. Record the start and stop times and the temperature on the bench sheet in TALS. The analyst will verify that a purple color persists or a black precipitate is present after the thirty minutes of heating. If this is not true, the digestion must be repeated using a smaller sample aliquot.

10.3.12 Allow the samples and standards to cool at room temperature.

10.3.13 Add 6 mL of sodium chloride-hydroxylamine hydrochloride solution to reduce the excess permanganate. Verify that the volume is at 50 mL.

10.3.14 For samples aliquoted using the Incremental Sampling Method combine the 5 individual sample cups for each sample and QC into one marked 250 mL container.

10.4 Calibration

10.4.1 All calibration standards are digested together with samples, as described in Section 10.3, prior to analysis. Preparation of calibration standards is described in Section 7.12.

10.4.2 Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.4.3 Detailed information regarding calibration models and calculations can be found in Corporate Policy CA-Q-P-003, *Calibration Curves and the Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

10.4.4 Initial Calibration (ICAL)

10.4.4.1 Calibration must be performed daily (every 24 hours) and each time the instrument is set up. The instrument calibration date and time must be included in the raw data.

10.4.4.2 Calibrate using six standards and a blank (see Section 7.12).

10.4.4.3 The calibration curve must have a correlation coefficient (r^2) ≥ 0.995 or the instrument shall be stopped and recalibrated prior to running samples. Sample results cannot be reported from a curve with an unacceptable correlation coefficient. The calibration curve is processed with an unweighted linear regression.

10.4.4.4 Record the microAbsorbance (µAbs.) for the 10 ppb standard

in the instrument maintenance log.

NOTE: It is not acceptable to reject calibration points for this method.

10.4.5 Initial and Continuing Calibration Blanks

10.4.5.1 An initial calibration blank (ICB) is tested immediately after the daily ICAL standards.

Acceptance Criteria: Absolute values for the calibration blanks must be less than ½ the standard RL. Client specific requirements take precedence. For example, DOD QSM 5 requires control of blanks to a concentration less than or equal to the LOD.

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.5.2 Continuing calibration blanks (CCBs) are run after every 10 samples and at the end of the run.

Acceptance Criteria: The absolute value of the blank result must be less than ½ the reporting limit. As just noted, DOD QSM 5 requires that results for blanks must be less than the LOD (refer to special project requirements).

Corrective Action: If the blank acceptance limit is exceeded, the analysis should be terminated, the source of contamination identified, and the instrument recalibrated.

10.4.6 Initial Calibration Verification (ICV), 4.0 µg/L (Hg Soil ICV)

The accuracy of the calibration standards is verified by testing a second source standard (ICV).

Acceptance Criteria: The ICV recovery must be within 90 - 110%.

Corrective Action: If the ICV acceptance limit is exceeded, the analysis should be terminated, the accuracy of the calibration standards checked, and the instrument recalibrated.

10.4.7 Reporting Limit Check Standard (CRA), 0.2 µg/L (Hg Soil RL)

The accuracy of results at the reporting limit is verified by testing a standard in every analytical run that is prepared at the reporting limit concentration.

Acceptance Criteria: The results for this standard must be within 50% of the expected value (20% for QSM 5.1).

Corrective Action: If the RL check acceptance limit is exceeded, the analysis should be terminated, the instrument operation checked, the instrument recalibrated, and associated samples reanalyzed.

10.4.8 Continuing Calibration Verification (CCV), 5.0 µg/L (Hg Soil CCV)

Calibration accuracy is monitored during the analytical run through the analysis of a known standard after every 10 samples and at the end of the run.

Acceptance Criteria: The CCV recovery must be within 80 - 120% except for QSM 5.0 where the CCV recovery limits are 90 - 110%.

Correction Action: Sample results may be reported only when bracketed by valid CCV pairs. If a mid-run CCV fails, the CCV may be reanalyzed once without modification to the instrument's operating conditions. If the reanalyzed CCV is found to be in control, the CCV analysis must be repeated with successful results or the analysis must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. If the cause of the CCV failure was not directly instrument related, the associated samples must be re-prepared and reanalyzed.

10.4.9 CCV Acceptance Criteria for sample run under a DOD QSM 5.0 program

CCVs must have a percent recovery of 90 - 110%. If the CCV fails the following options are available: Recalibrate and reanalyze all affected samples since the last acceptable CCV or immediately (within an hour) analyzed two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate then reanalyze all affected samples since the last acceptable CCV.

10.5 Sample Analysis

10.5.1 Set up the instrument and autosampler according to the manufacturer's

instructions.

10.5.2 Allow the samples to cool to room temperature prior to analysis or a decrease in the response signal can occur.

10.5.3 Pipet 10 mL of each sample and calibration standard into a disposable test tube for analysis

10.5.4 Analyze the standards and samples according to the manufacturer's instructions.

10.5.5 All measurements must fall within the defined calibration range to be valid. Dilute and reanalyze all samples with mercury concentrations that exceed the highest calibration standard.

NOTE: The instrument auto-dilutes samples. Any samples that require greater than a 10x dilution MUST be diluted manually.

10.5.6 If the sample results are negative and the absolute value is greater than the reporting limit, the sample must be reanalyzed.

10.5.7 Baseline correction is acceptable as long as it is performed after every sample or after the CCV and CCB.

10.5.8 The analytical sequence listed below must be followed. Refer to *Quality Control* Section 9.0 for quality control limits.

Instrument Calibration
ICV
ICB
CRA
CCV
CCB
Maximum of 10 samples
CCV
CCB
Repeat sequence of 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB

NOTE: Samples included in the count between CCVs include the method blank, LCS, MS, MSD, and field samples.

10.5.9 Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.0 Calculations / Data Reduction

Detailed calibration equations can be found in the corporate Policy CA-Q-P-003, *Calibration Curves and Selection of Calibration Points*, and under the public folder, *Arizona Calibration Training*.

11.1 Accuracy

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{spike concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spike concentration}} \times 100$$

11.2 Precision (RPD)

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3 Concentration = mg/kg or L $= \frac{C \times V \times D}{W}$

Where:

C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams or mL)

Note: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4 Documentation and Record Management

The following documentation comprises a complete CVAA raw data package:

11.4.1 Sample data entered into the preparation batch in TALS, which includes the batch number, list of samples, preparation analyst and date, identification of reagents and standards used, and identification of all measuring equipment used (e.g., balances, thermometers, pipettes).

11.4.2 Raw data (direct instrument printout as a PDF) with the analyst name and all required calibration information.

11.5 Reporting

11.5.1 Standard units for reporting solid sample results are mg/kg.

11.5.2 If dilutions are required due to insufficient sample, interferences, or other problems, the reporting limit and MDL are multiplied by the dilution factor,

and the data may require flagging.

11.5.3 Solid samples are reported on a dry-weight basis unless otherwise requested by the client. Reporting limits are adjusted for both sample size and percent solids.

11.5.4 All associated data are entered or uploaded into TALS as required.

11.5.5 Unless special instructions indicate otherwise, samples less than the reporting limit are reported as ND.

11.5.6 The initial data review is performed by the analyst while the second-level data review is performed by the area supervisor or designee. Both reviews are documented in TALS. See SOP DV-QA-0020 for more detail on the review process.

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. An initial method detection limit study is performed in accordance with Policy CA-Q-S-006. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency. MDL verifications are performed quarterly.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require such verification. A blank matrix is spiked at 1 - 2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for

each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee) the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, order chemicals based on quantity needed, and prepare reagents based on anticipated usage and reagent stability).

14.0 Waste Management

All waste will be disposed of in accordance with Federal, State, and local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this procedure, the policies in Section 13, *Waste Management and Pollution Prevention*, of the Corporate Environmental Health and Safety Manual, and Policy DV-HS-001P, *Waste Management Plan*.

14.1 The following waste streams are produced when this method is carried out:

14.1.1 Aqueous Acidic (Metals) - Corrosive - (J)

14.1.2 Expired reagents and standards – Contact the Waste Coordinator.

NOTE: Radioactive, mixed waste and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Final Update II, Revision I, September 1994, Method 7471A (Mercury).

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, February 2007, Method 7471B (Mercury).

15.3 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013.

15.4 Department of Defense Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Version 5.2, 2018.

15.5 U.S.EPA Statement of Work for Inorganic Analysis, ILMO3.0

16.0 Method Modifications:

Item	Method	Modification
1	7471A	An additional QC analysis, RL verification, is added
2	7471A	Methods 7470A and 7471A state that working standards "should be prepared fresh daily." The laboratory frequently prepares up to three batches of mercury samples, including digested calibration standards, each day. The third batch is typically prepared and digested late in the day, and then is analyzed the morning of the next day. The laboratory had developed the following information demonstrating that analysis within 24 hours, but on the second calendar day from preparation produces reliable results and is acceptable to the EPA: • Successful proficiency testing (PT) results for samples that were prepared and analyzed within 24 hours, but on successive days • Successful analysis of true NIST mercury standards within every analytical batch; and • A written comment from the EPA MICE Hotline stating that, with the supporting lab data, their opinion was that the laboratory's practice is "within the letter of the method as written."
3	7471A	Chapter 1 of SW-846 specifies the use of reagent water with a purity equivalent of ASTM Type II water. This SOP specifies the use of a Millipore DI system or equivalent to produce reagent water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.
4	7471A	Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of

Item	Method	Modification
		interest at or above ½ the reporting limit.
5	7471B	Method 7471B uses reagent water for the method blank. TestAmerica Denver is currently using glass beads when required.
6	7471B	Section 11.1 requires 50 mL of reagent water to be added to the sample with 15 mL of Potassium permanganate. TestAmerica Denver utilizes digestion tubes which do not allow for 50 mL of reagent water. 19 mL of reagent water is currently being added.
7	7471A	Section 7.1 specifies triplicate 0.2 g portions of sample for solids analysis. TestAmerica Denver instead utilizes a 0.5 - 0.6 g weight range in order to avoid targeting of specific weights and to comply with the requirements of the most recent method revision.

17.0 Attachments

Attachment 1: Summary of Quality Control Requirements
Attachment 2: MSA Guidance
Attachment 3: Instrument Maintenance
Attachment 4: Troubleshooting Guide

18.0 Revision History

- Revision 13 dated 19 September 2019
  - Updated standard concentration and spiking amount in Section 7.13.

- Revision 12 dated 31 May 2019
  - Annual technical review.
  - Minor formatting and language corrections throughout
  - Removed Section 3.1 due to redundancy
  - Updated amount of reagent water added in Section 10.3.5 to 3 mL.
  - Added QSM 5.1 CRA criteria to Section 10.4.7 and Attachment #1.
  - Removed Section 11.4.3 regarding data checklists
  - Removed reference to QSM 4.2 and added reference to QSM 5.2 in Section 15

- Revision 11 dated 31 May 2018
  - Annual Technical review.
  - Updated the spike amount used in Section 10.3.4 to 2ml.

- Revision 10 dated 31 May 2017
  - Updated the spike concentration for the LCS, MS and MSD in sections 7.16 and 7.17
  - Updated true value for LCS and MS/MSD in sections 9.4 and 9.5
  - Added DoD requirement of LCSD if no MS/MSD to sections 9.4 and 9.5
  - Added clarification to section 9.5 regarding parent and MS/MSD samples that are “over range”

- Revision 9 dated 01 Feb 2017

- Annual Technical Review
- Added a paragraph to Section 6.0 regarding the documentation of equipment IDs
- Updated Section 7.4 to make the aqua regia in 100 mL container
- Added Note 2 to Section 9.5 Referencing MS/MSD Policy Memoranda
- Added a paragraph to Section 10.3 regarding the documentation of equipment IDs
- Updated language in Section 12.1 regarding MDL studies
- Added current Section 12.2 defining LOQVs

- Revision 8 dated 28 Feb 2016
  - Annual Technical Review
  - Added current Section 3.1
  - Removed reference to AFCEE program in Section 9.3
  - Modified/Updated Section 9.5 – Corrective Actions for MS/MSD failures
  - Updated Sections 12.2 & 12.3 to be more consistent with other SOPs
  - Archived revision histories 2010 and earlier – available upon request

- Revision 7 dated 28 Feb 2015
  - Annual Technical Review
  - Removed reference to purging sample headspace from Section 4.4, outdated.
  - Section 4.6 – Removed reference to method 245.6
  - Changed MSDS to SDS
  - Removed references to "Cetac Only"
  - Added Section 7.5.3 regarding recording of data in TALS
  - Added TALS reagent IDs to standard names
  - Corrected concentrations of Cal Stds
  - Corrected Section references
  - Changed units of RL std to water units
  - Changed minimum sample volume to 10 g to accord with corporate policy
  - Added DoD V5 references
  - Changed sample aliquot to 0.5 – 0.6 g
  - Deleted references to adjust volume with 1% HNO3
  - Updated temperature of digestion to 95 ± 3 oC
  - Deleted Section 10.4.4.3 (Redundant)
  - Changed "counts" in Section 10.4.4.4 to microAbsorbance
  - Removed "Approximately" from ICV true values
  - Removed GC references from Section 10.4.9
  - Removed references to "resloping"
  - Added initial CCV/CCB pair to sequence

- Revision 6 dated 28 Feb 2014
  - Annual Technical Review
  - Removed references to Serial Dilutions and Post Digestion Spikes
  - Section 10.4 for incremental sampling was merged into Section 10.3
  - Updated Section 10.4.4.1 and 10.4.4.2 to note DOD QSM 5 criteria
  - Updated Section 10.4.7 to note DOD QSM 5 CCV criteria is 90-110.
  - Updated Attachment 2 for ICB,CCB and CCV criteria to DOD QSM 5
  - Added Attachment 5 for Troubleshooting

- Revision 5 dated 15 July 2013
  - Annual Technical Review

- Correction to formatting
- Changed reference to Standards Log to Reagent Module in the LIMS
- Added General Analytical Terms information to definition section
- Edited section 7.6, 10.4.8, 12.1 & 12.2 to reflect current practices.
- Changed RL reference in sections 7.18, 10.5.6, 10.6.8 and Attachment 2 to CRA
- Removed bullet point 5 under 11.5
- Removed Attachment 3 and renumbered the subsequent attachments
- Corrected references date for section 15.2
- Added Texas TRRP to section 12.1

- Revision 4 dated 30 September 2012
  - Clarified the language in Section 9.4 to be one LCS per batch.
  - Modified Section 7.16.2 to refer to Section 10.4.4 for additional detail.

- Revision 3.2 dated 13 July 2012
  - Updated Sections 7.6 and 7.7 to state Hg grade reagents are used
  - Updated Sections 10.4.11 to include a note about bringing samples to a final volume before the sample is mixed
  - Updated Section 10.5.4 and Attachment 2 to control calibrations blanks to ½ the RL.
  - Added Sections 10.5.3.5 to record the number of counts for the 10 ppb standard
  - Formatting and grammatical changes throughout

- Revision 3.1 dated 03 February 2012
  - Changed references of Multi-Incremental Sampling to Incremental Sampling throughout document
  - Annual Technical Review
  - Section 1.3 Added Incremental Sampling Method statement to SOP
  - Added introductory statement to section 7.0 regarding reagent purity
  - Updated section 9.5 and Attachment 2 for method blank control criteria
  - Section 10.2 Added Incremental Sampling Method preparation amount
  - Section 10.2.12 Added Incremental Sampling Method combination procedure
  - Section 10.2.13 Added Incremental Sampling Method final volume
  - Added dilution note to Section 10.3.5
  - Updated section 12.0 to reflect current laboratory practices

Earlier revision histories have been archived and are available upon request.

Attachment 1

Summary of Quality Control Requirements

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
ICB	Immediately following ICAL	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
ICV	Following ICB	90 - 110% recovery	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
RL Check Standard (CRA)	Following the ICV	50 - 150% recovery 80 - 120% for QSM 5.1	Terminate analysis; correct the problem; recalibrate or re-prepare and reanalyze batch.
CCV	Every 10 samples and at the end of the run	80 - 120 % recovery. 90 - 110% for QSM 5.0	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
CCB	Immediately following each CCV	Absolute value < ½ RL (< LOD for QSM 5.0)	Terminate analysis; correct the problem; recalibrate and rerun all samples not bracketed by acceptable CCVs or re-prepare and reanalyze batch.
Method Blank	One per sample preparation batch of up to 20 samples.	Project specific or ≤ ½ RL Sample results greater than 10% the blank concentration are acceptable.	Redigest and reanalyze samples. Note exceptions under criteria section.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 80 - 120%	Terminate analysis; correct the problem; redigest and reanalyze all samples associated with the failed LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	Recovery must be within statistical control limits, not to exceed 75 - 125%	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added.
Matrix Spike Duplicate	See Matrix Spike	Recovery within statistical control limits, not to exceed 75 - 125% recovery or in-house control limits; RPD ≤ 20%	See Corrective Action for Matrix Spike.

Attachment 2

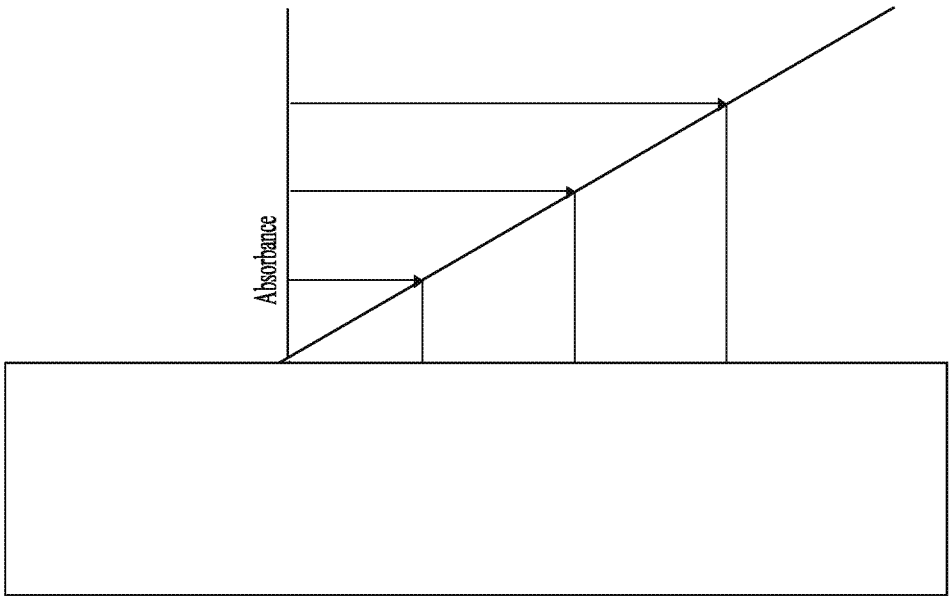
MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked aliquots should be the same (i.e., the volume of the spike added should be negligible in relation to the volume of sample).

To determine the concentration of analyte in the sample, the absorbance (or response) of each solution is determined and a linear regression performed. On the vertical axis the absorbance (or response) is plotted versus the concentrations of the standards on the horizontal axis using 0 as the concentration of the unspiked aliquot. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown. Calculate the correlation coefficient (r) and the x-intercept (where y=0) of the curve. The concentration in the digestate is equal to the negative x-intercept.

Figure 1



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration.

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

Attachment 3

Instrument Maintenance

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs, record the date, time and instrument number, then identify the problem and corrective action in the maintenance log. When the instrument is returned to service, record the return to service, the date, and any tests performed to verify proper operation.

The following preventative maintenance procedures are required to ensure that that the instrument is fully operational.

Cold Vapor Atomic Absorption

Daily	Monthly	Annually
Change rinse solution.	Check Hg lamp intensity.	Change Hg lamp.
Optimize light path.		Check liquid/gas separator.
Check argon flow.		
Check tubing. Replace as needed.		
Check drain.		
Check condition of dryer		

Attachment 4

Troubleshooting Guide

Problem	Possible Cause
Poor or No Absorbance or Sensitivity Check failed	Incorrect wavelength Dirty windows Window loose Etched or dirty optics Bad lamp Not enough or no sample introduced Empty sample cup Incorrectly made standards Gas leak
Erratic Readings	Source lamp not aligned properly Lamp not pre-warmed Injection tip partially clogged Contaminated reagents Contaminated glassware Drying tube saturated Bad lamp Injection tip hitting outside of tube Injection tip coated or not set properly Leak in sample tubing Power fluctuations Air bubbles in tubing
Standards reading twice or half normal absorbance or concentration	Incorrect standard used Incorrect dilution performed Dirty cell

1.0 PURPOSE

This Standard Operating Procedure (SOP) has been developed to provide field personnel with a structured methodology for obtaining general soils classifications. Classification of soils is necessary for geotechnical engineering analyses and for determining safety requirements for excavation and trenching operations (i.e., side-slope, benching, and shoring controls). This SOP is not intended to provide the protocol for geotechnical engineering or geological investigations.

For a more detailed field classification of soils, the procedures prescribed by American Society for Testing and Materials (ASTM) Test Method D-2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)* should be followed.

2.0 TERMS AND DEFINITIONS

Cohesive Soils: Soils having a major fraction of clay size particles with plastic properties. Cohesive soils exhibit strong cohesive properties due to inter-molecular bonding (plasticity) but are subject to significant deformation under load, i.e., consolidation. Cohesive soils are unsuitable for use as structural backfill. However, due to their plastic and relatively impermeable nature, they are suitable for liner and cap materials to minimize moisture infiltration (e.g., landfills).

Deleterious Material: Undesirable organics, trash, coarse inclusions, roots or other debris present in structural backfill material.

Grain-Size Distribution: A tabular and/or graphical presentation showing the various grain-size fractions of fine and course size soil particles and their percentages (based on total mass). The grain-size distribution is obtained from a grain-size analysis using various sized sieves and it used for soils classification.

Granular Soils: Soils having a major fraction of silt, sand, gravel, cobbles, or boulders (course particles). Granular soils vary in quality according to their grain-size distribution and organic content and they do not possess cohesive properties. They are generally acceptable for use as structural backfill material.

Plastic Index (PI): Parameter used to classify soils based on laboratory tests. The PI quantitatively measures the plasticity of a soil. The higher the plastic index, the more unsuitable a soil is for structural backfill, i.e., the soil will be subject to deformation, or consolidation, under load.

Plasticity: A term used to describe a soils ability to behave like a plastic material under pressure (i.e., deform or flow without cracking, separating, or crumbling). Generally, only clayey soils are considered to be plastic and the level of plasticity is variable based on the type of clay (chemical nature) and moisture content.

Soils Classification: A system used to define soil properties and characteristics. These systems are generally based on grain-size distribution and the plastic index of soils. The Unified Soil Classification System (USCS) is the standard system used for all Department of Defense (DoD) work.

Unified Soils Classification System (USCS): The USCS classifies soils based on grain-size distribution and the plastic index. This system classifies and identifies soils using abbreviations (e.g., C = Clay, S = Sand, M = Silt, etc.). It is the DoD accepted system used to specify soil types.

3.0 BACKGROUND

Formal soils classification systems have been developed by several organizations. The commonly accepted systems are the American Association of State Highway Transportation Officials (AASHTO) system, the US Department of Agriculture (USDA) Textural Classification system, and the *Unified Soil Classification System*. These systems classify soils based on grain size distribution, liquid limit, and the plasticity of the soil. The Occupational Safety and Health Administration (OSHA) Construction Standards (29 CFR Subpart P – Excavations, 1926.652) also presents a soils classification system for determining the required protection system for excavations. The OSHA system is based, in part, on the USDA and USCS.

For purposes of this SOP, the USCS will be used when soils are classified for engineering properties and evaluation. The USCS system is the accepted soils classification method by DoD agencies. The OSHA classification system will be used when classifying soils for determination of excavation safety requirements.

4.0 SOIL TYPES

In general terms, soil is classified as either non-cohesive (granular) or cohesive (non-granular). Granular soil particle size classifications include (with increasing particle size) fines (silts and clays), sands, gravels, cobbles, and boulders. Clay (cohesive) soils are fine grained particles with a chemically affinitive bond, i.e., cohesion. Silt soils are also fine-grained but do not possess a chemical affinity for intermolecular bonding.

Soils may also contain some fraction of organic material. Loam (a.k.a. topsoil) contains a significant fraction of organic material. Peat is a highly organic soil composed primarily of vegetable tissue in various stages of decomposition, peat is dark in color (brown to black) with a strong organic odor. Deleterious materials include roots, stumps, trash, or other non-natural debris.
Examples of *granular* soils include:

- Clean sands
- Sands and gravel (and cobbles, boulders)
- Angular gravel
- Silty sand
- Sandy silt
- Loamy sand

Examples of *cohesive* soils include:

- Clays
- Silty clays
- Clayey silts
- Sandy clay
- Clayey sands
- Clay loam
- Silty clay loam (in some cases)
- Sandy clay loam (in some cases)

5.0 SAMPLING

Note: For sampling soils to be analyzed for chemical compounds, refer to ECC SOP E-801, Soil Sampling.

5.1 In-Place

Several methods can be used to collect in-place soil samples including:

- Hand collection (borrow-pit, open excavation, test-pit, etc.)
- Hand and machine operated augers (ASTM D-1452)
- Drilling rig split spoon sampler (ASTM D-1586)

The sample collected must be representative of the stratum from which it was obtained. The sample shall be placed into a moisture-proof container (plastic bag, bucket w/ lid, etc.), labeled, and sealed to minimize moisture loss.

The number of samples necessary for characterization is dependent on the heterogeneity of the soils. A minimum of one grab or composite sample will be collected for each uniquely identifiable type of soil. Unless the volume of soils to be sampled is small (less than 50 cubic yards), a composite soil sample will be collected.

The sample will be given a unique identification (ID) and the location (aerial) and depth (stratum) will be recorded in the appropriate field-log.
An example of a sample ID is:

XXXX-LLLL-DDD-0001, where:

XXXX = project number
LLLL = general sample location (e.g., southeast area of site, etc.)
DDD = sample depth (bgs)
0001 = consecutive sample number

The sample will be sent to the geotechnical testing lab with a completed chain-of-custody indicating the date collected, sample ID, required test(s) and ASTM test method(s).

For detailed sampling procedures by auger or split-spoon sampling methods, refer to ASTM Test Method D-1452, *Practice for Soil Investigation and Sampling by Auger Borings* and ASTM D-1586, *Test Method for Penetration Test*

5.2 Stockpile

Unless otherwise specified, a minimum of one composite sample will be collected from a stockpile. Samples will be collected from a minimum of 4 locations from the stockpile. Sample methods are as follows:

- If a loader is available, take 3 to 6 bucket-loads of soil from each sample location at mid-height of the stockpile, place them into a small pile, mix by turning pile several times, and back-blade the pile. The sample is then collected from the center of the back-bladed area. Repeat this procedure until all samples are collected for compositing.
- If a loader is not available, hand-excavate (shovel) at the designated sample location at mid-height of the stockpile. Remove top 6 to 12 inches of soil and collect several shovels full of soil, place into small pile and mix by turning pile several times. Repeat this procedure until all samples are collected for compositing.

Each sample location must be documented in the appropriate field-log using a field-sketch of the stockpile. Prepare and label sample in accordance with procedures in previous section.

6.0 TESTING

In general, the following ASTM test methods are used to test and classify soils:

- ASTM D-420, *Standard Guide to Site Characterization for Engineering, Design, and Construction Purposes*
- ASTM D-422, *Standard Test Method for Particle-Size Analysis of Soils*
- ASTM D-1140, *Test Method for Amount of Material in Soils Finer than the No. 200 Sieve*
- ASTM D-2487, *Classification of Soils for Engineering Purposes (Unified Soils Classification System)*
- ASTM D-2488, *Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)*
- ASTM D-4318, *Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils*

Test methods D-422 and D-4318 are the minimum required tests for classification of soils. ASTM method D-2487 is the procedure by which the results from the D-422 and D-4318 tests are used to classify the soils.

7.0 CLASSIFICATION

7.1 Field

Field classification of soils will be completed in accordance with ASTM D-2488. The referenced test procedures will be completed by a geologist or civil/geotechnical engineer. The field classification method should only be used when sample collection and laboratory analysis are not practical, or, during geotechnical or geological investigations where immediate classification and/or supplementary (to laboratory) classification is useful. Following is a description of the more useful manual field tests:

- **Plasticity test** is used to determine if a soil is cohesive or cohesionless.
- **Dry strength test** is used to determine if a soil is cohesive and if a cohesive soil is fissured.
- **Thumb penetration test** is used to estimate the unconfined compressive strength of cohesive soils.
- **Drying test** is used to differentiate between cohesive soil with fissures, unfissured cohesive soil, and granular soil.
- **Other tests** include use of a pocket penetrometer or a hand-operated shearvane to estimate the unconfined compressive strength.

Refer to ASTM D-2488 or OSHA 29 CFR 1926 Subpart P, Appendix A for detailed manual test procedures and interpretation of results.

7.2 Engineering / Laboratory

Engineering classification of soils will be completed in accordance with ASTM D-2487. This method requires soil testing by a geotechnical/soils laboratory, sample collection will be completed in accordance with Section 4.0 by field personnel (QC personnel, sample technicians, etc.).

The geotechnical laboratory will complete, at a minimum, test methods ASTM D-422 and D-4318. Based on these test results, the laboratory will classify the soils and provide a report that presents the test results and the corresponding soil classification (ASTM D-2487). Additional tests may be prudent, as determined by the geotechnical engineer or geologist, based on the nature of the soils and the intended use of the analytical results.

7.3 Excavation Safety

As standard practice, soils classifications for excavation safety purposes will be completed in the same manner as the engineering classification. However, based on changing field conditions it may be necessary to complete field assessments without performing lab testing. Field classification of soils will be completed by a "competent person" as defined by OSHA. The competent person on a particular ECC project may be the project manager, quality

control officer, health and safety officer, or site superintendent, as appropriate.

The following field classification is based on the OSHA 29 CFR Standard 1926 Subpart P, Appendix A:

Type A Soils:

- Cohesive soil (see Section 3.0) with an unconfined compressive strength of 1.5 tons per square foot (tsf) or greater; and,
- Cemented soils such as caliche and hardpan; and,
- Soil cannot be fissured (i.e., tendency to break along planes or tension cracks at surface); and,
- Soil is not subject to vibration from traffic, pile driving or similar effects; and,
- Soil has not been previously disturbed; and,
- Soil is not part of a sloped, layered system where the layers dip into the excavation on a slope of 4H:1V or greater; and,
- Soil is not subject to other factors that would create a less stable condition.

Type B Soils:

- Cohesive soil (see Section 3.0) with an unconfined compressive strength greater than 0.5 tsf but less than 1.5 tsf; or,
- Granular cohesionless soils (see Section 3.0);
- Previously disturbed soils (not classified as Type C);
- Soil that meets the unconfined compressive strength or cementation requirements for Type A, but is fissured or subject to vibration; or,
- Dry rock that is not stable; or,
- Material that is part of a sloped layered system where the layers dip into the excavation on a slope less steep than 4H:1V, but only is the material would otherwise be classified as Type B.

Type C Soils:

- Cohesive soil with an unconfined compressive strength of 0.5 tsf or less; or,
- Granular soils including gravel, sand and loamy sand; or,
- Submerged soil or soil from which water is freely seeping; or,
- Submerged rock that is not stable; or,
- Material that is part of a sloped layered system where the layers dip into the excavation on a slope of 4H:1V or steeper.

Refer to SOP E-502 for excavation safety requirements and protocol.

8.0 RECORDS

Documentation shall be maintained at the job site per SOP requirements, and after project completion, with project files as specified in ECC Project Administrative Requirements. The original documents shall be maintained with the project files, unless the client specifically requests them. Copies of the field reports and tabulated test results will be included in the project report, but may also be submitted separately as specified in the submittal registry.

9.0 REFERENCES

1. OSHA Construction Standards, 29 CFR Subpart P – Excavations, 1926.652
2. ECC SOP E-502, Excavation Control Mechanisms
3. ECC SOP E-504, Compaction Testing
4. ASTM D2487-00 Standard Classification of Soils for Engineering Purposes (Unified Soil Classification System)

10.0 ATTACHMENTS

I. Unified Soil Classification System
II. Standard Drill Log