Quality Assurance Project Plan
For Exploration Drilling in Alaska
Outer Continental Shelf, Chukchi Sea,
on the Mobile Offshore Drilling Unit
Polar Pioneer

Draft: January 2015

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Acronyms List

°C	degrees Celsius
°F	degrees Fahrenheit
ADCP	Acoustic Doppler Current Profilers
Ag	silver
Al	aluminum
ANIMIDA	Arctic Nearshore Impact Monitoring in Development Area
ANOVA	analysis of variance
As	arsenic
Ba	barium
bbl/h	barrel(s) per hour
Be	beryllium
BI	benthic infauna (sample identification code)
BOD_5	5-day biochemical oxygen demand
BOP	Blowout preventer
CA	SPI computer analysis
CAB	Chemistry and Benthos
cANIMIDA	continuation Arctic Nearshore Impact Monitoring in Development Area
CC	Drilling cuttings chemistry (sample identification code)
CCA	canonical correspondence analysis
CCV	continuing calibration verification
Cd	cadmium
CFR	Code of Federal Regulations
cm	centimeter
CoC	chain-of-custody
COMIDA	Chukchi Sea Offshore Monitoring in Drilling Area
Cr	chromium
CRM	certified reference material
CSESP	Chukchi Sea Environmental Studies Program
CTD	conductivity, temperature, depth (profiler)
Cu	copper
CVAAS	cold vapor atomic absorption spectrometry
CVAF	cold vapor atomic fluorescence
DC	drilling fluid chemistry (sample identification code)
DCM	dichloromethane
DDW	distilled deionized water
DL	detection limit

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Acronyms List (Continued)

DMR	Discharge Monitoring Report
DOC	demonstration of capability
DOT	(U.S.) Department of Transportation
DQI	data quality indicator
DQO	data quality objective
DU	duplicate
EB	equipment blank
EDD	electronic data deliverable
EMP	environmental monitoring program
FAAS	Flame Atomic Absorption Spectrophotometry
FB	field blank
FC	fecal coliform
Fe	iron
FIT	Florida Institute of Technology
FWS	Fairweather Science
g	grams
gal	gallons
GC	gas chromatograph
GC/FID	gas chromatography/flame-ionization detection
GC/MS	gas chromatography/mass spectrometry
GFAAS	graphite furnace atomic absorption spectrometry
GP	GP
GPS	global positioning system
H_2O_2	hydrogen peroxide
H_2SO_4	sulfuric acid
HCl	hydrochloric acid
$HClO_4$	perchloric acid
HF	hydrofluoric acid
Hg	mercury
HMIS	Hazardous Materials Identification System
HNO_3	nitric acid
HPLC	high performance liquid chromatography
HSE	Health, Safety, and Environment
IATA	International Air Transport Association
ICAL	initial calibration
ICC	independent calibration check

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Acronyms List (Continued)

ICP-MS	inductively coupled plasma/mass spectrometry
ID	identification
IS	internal standards
ISO	International Organization for Standardization
LC	water (liquid) chemistry (sample identification code)
LCL	lower control limit
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LOD	limit of detection
LOQ	limit of quantitation
LWI	local work instruction
m	meter(s)
MB	method blank
MDL	method detection limit
MeHg	methyl mercury
mg	milligram
mgd	million gallons per day
mg/kg	milligram(s) per kilogram
mg/L	milligram(s) per liter
µg/L	microgram(s) per liter
M-I SWACO	contractor to Shell
mL	milliliter(s)
MLLW	mean lower low water
MODU	Mobile Offshore Drilling Unit
MQO	measurement quality objectives
MRL	method reporting limit
MS	matrix spike
MSD	matrix spike duplicate
NA	not available
NAD	North American Datum
$Na_2S_2O_3$	sodium thiosulfate
nMDS	non-metric multidimensional scaling
NMFS	National Marine Fisheries Service
Ni	nickel
NIST	National Institute of Standards and Technology
NPDES	National Pollutant Discharge Elimination System

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Acronyms List (Continued)

NS	not spiked
NSC	North Slope Crude
OBS	optical backscatter turbidity sensor
OCS	Outer Continental Shelf
OPR	ongoing precision and recovery
OSI	organism sediment index
PAH	polycyclic aromatic hydrocarbons
PARCCS	precision, accuracy, comparability, completeness, sensitivity
Pb	lead
PCA	principal component analysis
PD	percent difference
PIV	pre-injection volume
PM	project manager
ppm	parts per million
POC	particulate organic carbon
Polar Pioneer	MODU Polar Pioneer
PV	sediment profile imagery plan view photograph (sample identification code)
QA	quality assurance
QADU	laboratory duplicates
QAM	quality assurance manager
QAPP	quality assurance project plan
QC	quality control
RL	reporting limit
RIS	recovery internal speed
ROV	remotely operated vehicle
RPD	relative percent difference
RSD	relative standard deviation
SAV	submerged aquatic vegetation
Sb	antimony
SC	sediment chemistry (sample identification code)
SDG	sample delivery group
Se	selenium
SHC	saturated hydrocarbons
Shell	Shell Gulf of Mexico, Inc.
SIM	selected ion-monitoring
SIS	surrogate internal standard

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Acronyms List (Continued)

Sn	tin
SOP	standard operating procedure
SP	Sediment Profile Imagery photograph (sample identification code)
SPI	sediment profile imaging
SPI RPD	redox potential discontinuity (for SPI analysis)
SPP	suspended particulate phase
SRM	standard reference material
St/Tr	sterane/triterpanes
s.u.	standard units
TAH	total aromatic hydrocarbons
TAqH	total aqueous hydrocarbons
TB	trip blank
TBD	to be determined
TEM	total extractable material
Ti	titanium
Tl	thallium
TOC	total organic carbon
TPH	total petroleum hydrocarbons
TransOcean	Transocean Offshore Deepwater Drilling, contractor to Shell
TSS	total suspended solids
TU_c	chronic toxic units
UCL	upper control limit
U.S.	United States
USCG	United States Coast Guard
USEPA	United States Environmental Protection Agency
V	SPI visual analysis
VOC	volatile organic compound
WBM	water based mud
WC	worm chemistry (sample identification code)
WET	whole effluent toxicity (testing)
ZGFAAS	Zeeman or Continuum background correction
Zn	zinc

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Chukchi Sea Permit No.: AKG-28-8100
QAPP Revision Summary Table

Revision Number	Revision Date	Revision Reason

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1.0 Project Management

1.1 Organization

This document serves as the Quality Assurance Project Plan (QAPP) for all requirements under the Permit No.: AKG-28-8100 National Pollutant Discharge Elimination System (NPDES) General Permit (New and Existing Sources and New Dischargers in the Offshore Subcategory of the Oil and Gas Extraction Category) (herein referred to as the GP) for wastewater discharges into Arctic Ocean Outer Continental Shelf (OCS) waters. Section 1 presents quality assurance and quality control information that is relevant to the larger permit requirements (i.e., including both operational, on-rig discharges and the environmental monitoring program (EMP). Section 2 presents quality assurance and quality control information that is specific to the operational, on-rig discharge monitoring permit requirements (e.g., Permit Section II.B). Section 3 presents quality assurance and quality control information that is specific to the EMP Plan of Study (e.g., Permit Section II.A.13a-n).

1.2 Purpose and Scope

This section identifies Shell Gulf of Mexico, Inc. (Shell) as the operator for the Chukchi Sea Exploration Program and holder of the GP for wastewater discharges into Arctic Ocean Outer Continental Shelf (OCS) waters.

This QAPP defines policies and systems governing the assessment and quality control of sampling, data collection, and laboratory analyses performed to document compliance with the GP. This QAPP will be used to assist in sampling and analysis required to support compliance with effluent limitations and monitoring requirements for operational discharges as well as planning for the collection and analysis of data required in the Environmental Monitoring Program (EMP) Plan of Study specified in the GP.

Development of the QAPP for work performed under the GP was based upon applicable guidelines and regulatory documents including:

- AKG-28-8100 National Pollutant Discharge Elimination System (NPDES) General Permit;
- Title 40 of the Code of Federal Regulations, Part 435 (40 CFR Part 435);
- 40 CFR Part 136; and
- U.S. Environmental Protection Agency (USEPA) regulations and guidance, including Requirements for Quality Assurance Project Plans (EPA/QA/R-5) (USEPA 2001) and Guidance for Quality Assurance Project Plans (EPA/QA/G-5) (USEPA 2002).

1.3 Project Definition/Background

In order to fulfill the requirements of the GP, field measurements and environmental samples will be collected as part of investigations and characterizations as specified in the EMP, and also to demonstrate compliance with effluent limitations and monitoring requirements specified for operational discharges.

This QAPP has been prepared to satisfy the requirements of Section IV.A. of the GP. This QAPP defines the quality assurance (QA) and quality control (QC) procedures that will be followed to ensure that the

data obtained from field and laboratory analyses are of known and acceptable quality to achieve project data quality objectives. The QC procedures outlined herein are intended to support the activities requiring the collection of data as described in the EMP, the Best Management Practices (BMP) Plan, written procedures, and this QAPP. This QAPP provides specific guidance regarding sample collection and handling procedures, referencing the Appendices to the BMP and written procedures, as appropriate.

General information about the vessel, support vessels, and exploratory well locations can be found in Section 3 of the BMP. Additional information about the Chukchi Sea site description can be found in the EMP.

1.4 Project Task Description

Shell has contracted Transocean Offshore Deepwater Drilling, (Transocean), the owner of the semi-submersible, MODU (Mobile Offshore Drilling Unit) Polar Pioneer (*Polar Pioneer*), for operations of the vessel during transit and while exploration activities are being conducted. The *Polar Pioneer* is an offshore oil and gas semi-submersible drilling vessel adapted for operation under Arctic conditions. The *Polar Pioneer* has all necessary drilling equipment and ancillary facilities to explore and complete exploratory wells in the Chukchi Sea.

Shell plans to drill six exploratory wells in the Burger Prospect of the Chukchi Sea. Each of the six possible drill sites will be permitted for drilling to allow for operational flexibility. The prospect location is depicted in Figure 3-1 of the BMP. Table 1-1 presents geographical drill site locations for each well in the drilling program.

Table 1-1 Geographical Drill Site Locations

Wells Covered by BMP Plan	Area	Lease Block (Surface)	Surface Locations (NAD 83)[1]		OCS-Y Number
			Latitude (N)	Longitude (W)	
Burger A	Posey	6764	N71°18’30.92”	W163°12’43.17”	OCS-Y-2280
Burger F	Posey	6714	N71°20’13.96”	W163°12’21.75”	OCS-Y-2267
Burger J	Posey	6912	N71°10’24.03”	W163°28’18.52”	OCS-Y-2321
Burger R	Posey	6812	N71°16’06.57”	W163°30’39.44”	OCS-Y-2294
Burger S	Posey	6762	N71°19’25.79”	W163°28’40.84”	OCS-Y-2278
Burger V	Posey	6915	N71°10’33.39”	W163°04’21.23”	OCS-Y-2324

Note:
[1] North American Datum 1983.

Shell has requested authorization for a number of discharges during drilling operations that are regulated by the EPA under the GP and by 40 CFR Part 435. Data collection to verify compliance with the effluent limitations and monitoring requirements of the GP will include, but is not limited to, visual assessment of receiving waters; discharge flow-rate and volume measurements; and collection of samples for rig compliance laboratory analysis and fixed subcontract laboratory analysis. Operational discharge compliance monitoring activities and QA/QC procedures are defined in Section 2 of this QAPP.

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In addition to operational discharge compliance monitoring activities, environmental samples will be collected as part of assessments and characterizations required by the EMP. Data collection activities and QA/QC procedures related to the EMP are described in Section 3 of this QAPP.

1.5 Project Quality Objectives

The overarching project objectives for Shell's exploration drilling program in the Chukchi Sea are to monitor effluent discharges, implement the EMP Plan of Study, and ensure the collection of data that are of sufficient type, quantity, and quality to meet the regulatory requirements specified by the GP.

1.5.1 Data Quality Objectives

The DQOs, details regarding the sampling design, QA/QC procedures, and data collection can be found in Section 2 for operational discharge compliance sampling and in Section 3 for the EMP.

1.5.2 Data Quality Indicators

The quality of the data to be collected for this project will be verified through appropriate criteria established for both sampling procedures and analytical methods. The criteria should relate to data quality indicators (DQIs) consisting of precision, accuracy, representativeness, comparability, completeness, and sensitivity (commonly referred to as PARCCS) parameters. The quality of the sampling procedures and laboratory results will be evaluated for compliance with project DQOs through a review of these parameters. Project DQOs will be considered met when the quality of the data meet precision, accuracy, representativeness, completeness, comparability, and sensitivity requirements specified in this QAPP.

Analytical DQOs will be evaluated by reviewing the QC parameters described in the following sections. Laboratory and field quality control samples are described in more detail in Section 1.10. DQIs that relate specifically to analyses required for the monitoring of operational discharges are included in Section 2. DQIs that relate specifically to analyses required for the EMP are included in Section 3.

1.5.2.1 Precision

Precision measures the reproducibility of measurements. Analytical precision is the measurement of the variability associated with duplicate (two) or replicate (more than two) analyses. Precision will be evaluated by comparing the following:

- Laboratory control sample (LCS) and LCS duplicate (LCSD) (if prepared and analyzed) to determine the accuracy and precision of the laboratory procedures and verify matrix interference by comparing to MS results;
- Matrix spike (MS) and matrix spike duplicate (MSD) samples to determine the effect of the sample matrix on the precision of the results generated using the selected analytical method;
- Primary and field duplicate sample results;
- Analysis of standard reference material (SRM); and/or
- Laboratory replicate analyses.

The LCS determines the precision of the analytical method. If the recoveries of the analytes in the LCS are within established control limits, then precision is within acceptable limits. In this case, the

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comparison is not between a sample and a duplicate sample analyzed in the same batch; rather, the comparison is between a given sample and those samples analyzed in previous batches. If an LCSD is prepared for the batch, the precision of the analysis can be evaluated for the batch.

Precision of two similar values is evaluated by calculating relative percent difference (RPD) using the following equation:

$$RPD = \frac{2|(D_1 - D_2)|}{D_1 + D_2} x\,100$$

Where: D_1 = first sample value
D_2 = second sample value (replicate)
RPD = relative percent difference

Percent difference (PD) is a measurement of precision as an indication of how a measured value is different from a "real" value. It is used when one value is known or certified, and the other is measured. The formula for calculated PD is as follows:

$$Percent\ \ Difference = \frac{X_2 - X_1}{X_1} x\,100$$

Where: X_1 = known value (e.g., SRM certified value)
X_2 = determined value (e.g., SRM concentration determined by analyst)

If two or more aliquots of the same sample are prepared and analyzed by the laboratory, then these are referred to as laboratory replicates. Precision of replicate samples is evaluated by calculating the RSD using the following equation:

$$\%RSD = \frac{\sqrt{\dfrac{\sum_{i=1}^{i=n}\left|D_i - \overline{D}\right|^2}{n}}}{\overline{D}} x\,100$$

Where: D_i = the individual sample concentrations
$\overline{D}$ = the mean of *n* values
n = the total number of values

Note: Report the absolute value of the result. The RSD is always positive.

1.5.2.2 Accuracy

Accuracy is a statistical measurement of correctness and includes components of random error (variability due to imprecision) and systemic error (variability that can be assigned to a specific component of the measurement process). Random errors and systematic errors reflect the total error associated with a

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measurement. A measurement is accurate when the value reported does not differ from the true value or known concentration of the spike or standard.

Percent recovery is a measurement of accuracy, where one value is compared with a known/certified value. Analytical accuracy is measured by comparing the percent recovery of analytes spiked into an LCS or a MS sample to a control limit. Analysis of SRM may also be used to evaluate accuracy.

The formula for calculating percent recovery is as follows:

$$Percent\ Recovery = \frac{amount\ detected}{amount\ expected}\ x\ 100$$

1.5.2.3 Representativeness

Representativeness is a qualitative term that refers to the degree by which the data accurately and precisely depict the characteristics of a population, whether referring to the distribution of a contaminant within a sample, a sample within a matrix, or a contaminant at a site. Representativeness is the qualitative term evaluated to determine that measurements are made and physical samples collected at locations and in a manner that result in proper characterization of a matrix or media. Subsequently, representativeness ensures that a sampled population represents the target population and an aliquot represents a sampling unit.

Assessment of representativeness shall be achieved through use of the standard field, sampling, and analytical procedures. Representativeness will be evaluated by reviewing the following:

- Sample quantities and locations;
- Sampling procedures and equipment;
- Sample CoC forms, laboratory report forms, and laboratory records; and
- Holding times and preservation.

1.5.2.4 Comparability

Comparability addresses the degree to which different methods or data agree or can be represented as similar. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. Comparability is achieved by the following:

- Using standard methods for sampling and analysis;
- Reporting data in standard units;
- Normalizing results to standard conditions;
- Operating instruments within their calibrated range according to established procedures based on approved methodology; and
- Using standard and comprehensive reporting formats.

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1.5.2.5 Completeness

Completeness is calculated for the aggregate data for each analyte measured during any particular sampling event. Completeness is calculated and reported for each method, matrix, and analyte combination. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. For completeness requirements, valid results are defined as all results not rejected through data validation.

The following formula is used to calculate completeness:

$$\%\,\text{completeness} = \frac{\text{number of valid results for samples analyzed}}{\text{number of possible results for all samples}}$$

1.5.2.6 Sensitivity

Sensitivity is the ability of a method or instrument to detect the target analytes at the level of interest. Analytical methods are selected that will provide results with the sensitivity to meet the project DQOs. The laboratory detection limits and reporting limits will be evaluated against the permit limits in order to determine whether the analytical methods and/or laboratory meet the project DQOs.

1.6 Special Training and Certifications

Each contractor has specific corporate policies, training manuals/programs, safety & environmental risk assessments, and compliance tracking systems as it applies to all oil and gas exploration operations. The validity of collected data depends, in part, on the qualifications and training of the personnel involved with sampling and analysis. Trained, qualified personnel are required to perform field activities related to operational discharge and monitoring and the EMP. Specialized training or certifications required to complete project tasks related to sampling and analysis are included in the following sections.

1.6.1 Health, Safety, and Environment Program Training

Shell and its contractors have implemented health, safety, and environmental compliance programs that direct all aspects of the operation of the *Polar Pioneer* and employees who are assigned to the vessel. All assigned employees are provided regular training on these measures and programs. Aboard the vessel, management of all health, safety, and environmental compliance programs are implemented by Shell and its contractors. The detailed plans may be available for review aboard the *Polar Pioneer* if requested. Shell will have full time Health, Safety, and Environment (HSE) staff on board during operational periods.

1.6.2 General Permit Training Curriculum for M-I SWACO NPDES Discharge Compliance Specialists

The M-I SWACO NPDES Operational Discharge Compliance Specialists are responsible for maintaining copies of their training records. Specific training requirements related to operational discharges and monitoring activities are specified in the written procedures, which are incorporated by reference into the BMP and in Appendix A of this QAPP.

Each M-I SWACO NPDES Compliance Specialist shall participate in a regulatory training curriculum prior to providing GP compliance data. The curriculum includes presentations and written tests on the following elements:

- Regulatory requirements.
- QAPPs.
- Documentation procedures.
- Applicable written procedures.

Upon completion of the training curriculum, each trainee and trainer shall complete the appropriate M-I SWACO NPDES Compliance Specialist Training Documentation Form (M-I SWACO Form 2009-1, Appendix A). This form documents the employee's training on the QAPP, written procedures, specific equipment, analytical techniques, and QC requirements. NPDES Compliance Specialist training and the NPDES Compliance Specialist Training Documentation Form must be completed prior to performing any GP compliance duties. The M-I SWACO NPDES Compliance Supervisor will maintain the original forms in the employee training records and the employee maintains a copy within the M-I SWACO NPDES Compliance Specialist notebook.

1.6.3 M-I SWACO NPDES Discharge Compliance Specialist Demonstration of Capability

M-I SWACO NPDES Discharge Compliance Specialists who perform measurements, analyses, or evaluations, or who operate instruments or equipment shall have demonstrated proficiency for the specific procedures for which there is a mandatory demonstration of capability (DOC) requirement in the SOP. Demonstration of capability shall be performed each time there is a change in instrument type, personnel, or test method. Procedures for performing and documenting the demonstration of capability are discussed in SOP 1008 Demonstration of Capability (Appendix A). The Demonstration of Capability Certification Statement included in this procedure shall be completed for each analyst, matrix, and method after successful completion of the proficiency testing. One certificate may be used for multi-analyte methods.

1.7 Documents and Records

The quantity of supporting documentation required to conduct environmental analyses necessitates the establishment of a formal system for generating, checking, inventorying, and archiving documents. This section describes document control and recordkeeping as it applies to field and laboratory data generated to demonstrate compliance with the QAPP.

During the Chukchi Sea exploration drilling program, both hard copy and electronic records will be generated by numerous organizations involved in various aspects of the program. Each contractor to Shell is responsible for ensuring that the records generated by their field staff and/or subcontractors are in compliance with the requirements of this QAPP. All organizations will maintain original records in the specific locations required by their respective QA programs, and copies of their records will be provided as required during and following execution of the project. In general, documentation and recordkeeping will be conducted in accordance with this document (Sections 2.7 and 3.9).

Rig compliance laboratory records are discussed in following sections. The QA program of subcontract laboratories, including the Barrow mobile laboratory and fixed analytical laboratories, governs their

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document control. Maintenance, revision, approval, distribution, archival, and retention of records will be in accordance with the subcontract laboratory QA program.

1.7.1 Document Revisions

The QAPP and associated written procedures are controlled documents. Each time a revision is made to this manual, it shall be reviewed, documented, and approved. Whenever revisions are made, or addenda added to the QAPP, a document control system shall be initiated to confirm that all parties holding a controlled copy of the QAPP receive the revisions/addenda, and that outdated material is removed from circulation.

The following procedures will be implemented to ensure that project personnel have the current versions of these documents:

- The document includes a version number and effective date;
- Each organization will maintain a master list of current written procedures, which are approved by management and assigned a revision number and effective date;
- A distribution list is maintained for the QAPP; and
- Written procedures are reviewed and revised in accordance with each organization's internal QA program.

The QAPP shall be reviewed at least annually and will be revised or amended as substantive changes are warranted. As the QAPP is revised, the revision number on the appropriate pages is incremented and the modified pages are released to the distribution list. A summary of the revisions with the date and a description of the revision is maintained on the signature page. The QAPP revision shall be submitted for approval by the same authorities that performed the original review.

1.7.2 Rig Compliance Laboratory Documentation

Field personnel from each organization shall retain copies of observations, calculations and derived data, calibration records, results, and/or reports in accordance with the BMP and each organization's QA program. Field observations will be documented in real time in bound field logbooks and will provide a record of field activities, observations, and measurements during sampling. Details regarding field and laboratory documentation are included in Sections 2 and 3.

1.7.3 Archival, Maintenance, and Retention of Project Files

Central project files are maintained in accordance with each organization's internal QA program. The central project files consist of completed rig compliance laboratory documentation, as well as fixed laboratory reports, computer program documentation, training records, and qualified subcontractor files.

Pursuant to permit regulations, project files will be retained for a period of at least five years from the date of the sample, measurement, report, or application, whichever is longer. Records are maintained electronically and/or in hard copy. Records will be maintained, archived, and retained in accordance with Section 11.5 of the BMP.

Record retention includes, but is not limited to, the following:

- Laboratory reports;
- Chain-of-custody (CoC) forms;
- Original monitoring data, including logbooks, field forms, electronic data capture, or other records in which monitoring data are first documented;
- Logs or other documents used to record field measurements, such as flow meter readings; and
- Calibration and maintenance logs for field instruments and rig compliance laboratory equipment, as they relate to measurement of volume or monitoring quality.

1.7.4 Confidentiality and Proprietary Rights

Personnel may have access to confidential information concerning permittees operations and company operations. This knowledge may include expertise, technical information, technical software, and records related to operations, finance, accounting, sales, personnel, and management, policies, or other matters. Personnel may also have access to permittee and company trade secrets, including secret formulations, techniques, methods, processes, data, discoveries, developments, designs, improvements, inventions, and the like. The protection of these trade secrets and confidential information against unauthorized disclosure or use is of critical importance. Consequently, employees and contractors may be required to execute nondisclosure and confidentiality agreements.

Any confidential business information, defined as trade secrets and commercial or financial information obtained from a person and privileged or confidential, provided as part of voluntary and/or mandatory submittals to the EPA shall not be disclosed.

1.8 Sample Handling, Documentation, and Chain-of-Custody Procedures

This section includes sampling and data collection procedures and policies for ensuring compliance with requirements of the GP applicable to exploration drilling activities to be conducted by Shell in the Chukchi Sea.

An essential part of the sampling activities of any environmental project is assuring the integrity of the samples from collection through data reporting. Environmental analyses involve the collection and shipping of numerous samples from different sampling sites. Sample labels and CoC forms are used to document identification and handling of samples from the time of collection through the completion of chemical and/or physical analysis. Documentation of the history of a sample will be prepared and maintained to demonstrate that the data are a true representation of the environmental media. The CoC record is used to demonstrate that a sample was not tampered with or altered in any way that may bias the analytical accuracy of the laboratory results. It is extremely important that CoC records be complete, accurate, and consistent.

This QAPP governs field and analytical sample handling, documentation, and CoC procedures for samples analyzed in the field at the rig compliance and Barrow mobile laboratories and for samples transported to fixed subcontract laboratories. Additional details specific to the handling, documentation, and CoC procedures for the operational discharge compliance sampling program are included in Section 2; and for the EMP are included in Section 3.

The QA program of the fixed subcontract laboratory performing analyses governs laboratory sample control procedures.

1.8.1 Sample Containers and Preservation

Subcontract laboratories supply containers for fixed laboratory analyses. These containers are pre-cleaned and/or preserved in accordance with analytical method requirements. Details regarding the containers required for specific analyses can be found in Sections 2 and 3.

1.8.2 Sample Storage

Samples shall be stored in limited-access, temperature controlled areas while in the rig compliance laboratory or until packaged and shipped to the subcontract laboratory for analysis. In general, the acceptance criterion for sample storage is <6°C. Thermal preservation requirements for specific analyses are outlined in Sections 2 and 3.

1.8.3 Sample Retention and Disposal

Samples will be retained under proper storage conditions in accordance with the written procedures, and/or analytical methods. Analytical samples are retained and disposed of at the subcontract laboratories in accordance with the internal laboratory QA program.

1.8.4 Sample Labeling

As samples are collected and containerized, each sample is given a unique sample identification (ID) number. The sample ID is documented in the field logbook and on the CoC form. Sample IDs will be assigned in accordance with the specific procedures in Sections 2 and 3.

Each sample container must include the following information:

- Date - A six-digit number indicating the year, month, and day of collection, in this order;
- Time - A four-digit number indicating the military time of collection;
- Sample ID - A unique identification number which may contain the above information, but which distinguishes among samples collected from the same site;
- Preservative (if any);
- Sampler – Signature or initials of person collecting the sample; and
- Requested analyses.

Field QC samples (equipment blanks, field duplicates, etc.) are labeled as above, but are not identified as QC samples on the labels.

1.8.5 Chain-of-Custody Procedures

After collection, preservation, and identification, the sample is maintained under CoC procedures discussed in this section.

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Sample custody records are the administrative records associated with the physical possession and/or storage history of each individual sample from sample collection to the final analytical result and sample disposal. Sample custody will be initiated by the sample collection records that identify for each sample the unique sample ID, date, time, location, and collector.

CoC forms document sample collection and shipment to the laboratory. CoCs are legal documents that record the transfer and disposition of collected environmental samples. The CoC form will identify the contents of each shipment and provide objective physical evidence of the possession history and integrity of each sample from collection through analysis. Transfer of physical custody shall be recorded.

CoC procedures begin in the field with the collection and containerization of samples. A sample is considered under CoC if:

- The samples are in a person's possession, or
- The samples are in a person's view after being in that person's possession, or
- The samples were in a person's possession and then were locked or sealed to prevent tampering, or
- The samples are in a secure area.

As soon as each sample has been collected, containerized and labeled, it is entered on the CoC form. Each cooler should contain one CoC form representing all of the samples present in the cooler. The sampler shall complete the information on the form accurately and legibly. Any corrections to the CoC form must be made using a single line through the incorrect entry. Corrections must be initialed and dated by the person making the change.

The chain-of-custody form must include:

- Sample originator (client);
- Sample identification / location;
- Project identification;
- Date and time of collection;
- Collector's name (printed);
- Sample type (e.g., liquid or sediment);
- Analyses required;
- Preservation used; and
- Signatory of release/acceptance through transport.

Once they have been properly labeled and logged, samples are packaged for shipment and sent to the laboratory with a CoC record sealed into each cooler. Before the sample cooler is shipped, the sampler must sign and date the CoC form under "Relinquished by". The original CoC form accompanies the

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samples during shipment; a copy is maintained by the shipper. Coolers containing samples will be custody sealed prior to shipment to subcontract analytical laboratories.

When samples are received at the subcontract laboratory, the laboratory sample custodian signs the CoC form as "Received by," and enters the date and time. The sample custodian shall carefully inspect samples as described in the laboratory SOP, including:

- Intact air-tight seal,
- Intact CoC,
- Evidence of alteration or damage to samples or packaging, and
- Completeness of accompanying records.

The laboratory's sample receipt record shall explicitly state the condition, including the measurement of the temperature blank or cooler temperature, for each incoming sample. The Shell Environmental Compliance Engineer or his/her designee is notified of arrival and condition of each shipment, and of any discrepancies in accompanying paperwork, immediately after login inspection. After the sample log-in is complete, another copy of the CoC record, which includes laboratory sample numbers and notations of any discrepancies, is sent to the sampler identified on the CoC form and/or to the Shell Environmental Compliance Engineer, as instructed. The original CoC is filed in the laboratory, with the shipper's waybill or airway bill attached, and a copy of this record is provided to Shell.

Samples for rig compliance laboratory analyses shall be kept in a designated secure area. CoC forms are not required for rig compliance laboratory samples. Sample collection activities and identification of samples shall be recorded in field logbooks.

1.8.6 Packaging and Shipment of Samples

Samples shall be packaged and shipped in accordance with each organization's QA program and SOPs/LWIs. Standard procedures for packaging and shipment of samples are necessary for the following reasons:

1) To protect persons handling, receiving and unpacking shipped samples;
2) To minimize loss of samples through breakage or delays in shipment;
3) To document sample integrity; and
4) To comply with applicable U.S. Department of Transportation (DOT) (49 CFR) and International Air Transport Association (IATA) regulations.

Details regarding specific procedures for packaging and shipping samples are included in Sections 2 and 3.

Environmental samples are samples of drilling fluids or other media where contamination is expected to be in relatively low concentrations. For the purpose of this QAPP, environmental samples are those samples whose toxic, flammable, corrosive or otherwise hazardous constituents represent less than one percent by volume.

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1.9 Field and Laboratory QC Samples

The sampling designs for the operational discharge compliance sampling program and the EMP incorporate QC procedures and checks in both the field and laboratory to assess data quality. The following sections outline the most common types of field and laboratory QC samples that may be collected for the project. Sections 2 and 3 include specific types and frequencies of QC samples that will be collected and analyzed for operational discharge compliance sampling and the EMP. Section 3 also includes additional QC samples that will be collected for the EMP.

1.9.1 Field QC Samples

Field QC samples will be collected in the same type of sample containers and handled in the same manner as other field samples. The field QC samples will be assigned unique sample numbers and will be submitted to the analytical laboratory as routine samples. If nonconformances are detected in the results for field QC samples, the data associated with the QC samples will be evaluated to determine if the quality and/or usability of the data are affected.

1.9.1.1 Trip Blanks

Trip blanks are collected only for volatile organic compound (VOC) samples. A trip blank consists of an uncontaminated sample of similar matrix that is transported from the laboratory to the sampling site, handled like an environmental sample, and returned to the laboratory for analysis without having been exposed to sampling procedures (i.e., not opened in the field). The results are used to assess contamination introduced during shipping and field handling procedures. Trip blanks are prepared and analyzed at the frequency of one per shipping container of VOC samples.

1.9.1.2 Temperature Blanks

A temperature blank is a container of water that is packed and shipped to the laboratory with the field samples requiring preservation by cooling. Upon arrival of a cooler at the laboratory, the laboratory measures the temperature of the blank. This temperature reading is used to represent the conditions of the field samples during shipment to the laboratory. This information is used by both the laboratory and by the data reviewer. If the temperature blank exceeds the sample-specific thermal preservation criteria, the laboratory must notify Shell immediately for guidance.

1.9.1.3 Field Duplicates

A field duplicate is a generic term for two (or more) field samples collected at the same time in the same location. Field duplicate samples are submitted as blind samples to the laboratory and are taken through all steps of the analytical preparation and analysis process in an identical manner. These samples are used to assess precision of the entire data collection activity, including sampling, analysis, and site heterogeneity. Field duplicates shall be collected where measurable contamination is likely to be present. If contaminant levels in the duplicates are below detection limits, they cannot be used for data quality assessment. Field duplicates are collected at a frequency of 5% of the field samples (one field duplicate sample for every 20 or fewer field samples).

There are two categories of field duplicate samples defined by the collection method: co-located field duplicates and subsample field duplicates. Co-located field duplicates are two or more independent

samples collected from side-by-side locations at the same point in time and space so as to be considered identical. Co-locates are samples collected from adjacent locations, or water samples collected from the same sampling point at the same time that have not been homogenized. Subsample field duplicates samples are obtained from one sample collection at one sample location. The sample is homogenized and then subsampled in the field to form an original and duplicate sample.

Drilling fluid samples may contain gradients of chemical constituents. It is therefore necessary to thoroughly mix a quantity of drilling fluid and then split the resulting homogenized material into two separate sample containers to obtain identical field duplicate samples. The contamination risk is mitigated through the use of specially cleaned containers and properly decontaminated or disposable sampling equipment and implements for mixing and transferring the sample material.

1.9.1.4 Matrix Spikes and Matrix Spike Duplicates (MS/MSDs)

A matrix spike (MS) is performed by spiking one of a pair of duplicate samples with a known quantity of target analyte and extracting and analyzing both the spiked and the unspiked samples. In the absence of matrix interferences, the difference between the analytical results for these two duplicates will yield an acceptable recovery rate for the spike compound. If matrix interferences are present, their effect on the analytical results for the MS sample can indicate a similar effect on other samples of a similar matrix.

Matrix spikes may also be used to measure precision and accuracy of the sampling and analysis process by analyzing two spiked aliquots and one unspiked aliquot of a sample. This second spiked sample aliquot is called a MS duplicate (MSD). Accuracy is measured by calculating the difference between the measured spike concentration and the known concentration added to the MS and MSD. The closer the two measured concentrations are to the true concentration, the higher the level of accuracy in the analytical process. Precision is measured by calculating the difference between the two spike concentrations recovered in the MS and MSD. The greater the difference between the two recovered spike amounts, the lower the precision of the sampling and analysis process.

Because three separate aliquots, or portions, of sample have to be analyzed when performing an MS/MSD, it is often necessary to submit double or triple the volume of sample for an MS/MSD than that required for a normal sample. The MS/MSD samples are not separate samples, and the same sample number will be assigned to the primary (parent) sample and the extra volume supplied for the laboratory MS/MSD. CoC forms will indicate the sample designated for MS/MSD when additional material is provided. MS/MSD samples will be collected at a frequency of 5% of the field samples (one MS/MSD pair for every 20 field samples).

1.9.2 Laboratory QC Samples

Laboratory samples will be processed and analyzed in analytical batches or sample delivery groups (SDGs) of 20 or fewer field samples plus laboratory QC. A suite of QC samples that monitors the accuracy and precision of the methods will be incorporated into each batch; these samples are defined below. In addition to these QC samples, surrogate internal standards will be spiked into each sample analyzed for organic compounds. Laboratory QC and acceptance criteria for the specific analytical methods are included in Sections 2 and 3.

1.9.2.1 Method Blank

A method blank is an analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. A method blank is prepared and analyzed with every analytical batch. The method blank is carried through the complete sample preparation and analytical procedure and is used to assess possible contamination resulting from the analytical process.

The presence of analyte in a method blank at a concentration exceeding the analytical data quality objective indicates the need for further assessment of the data. The source of contamination should be investigated, and measures must be taken to correct, minimize, or eliminate the problem. No analytical data shall be corrected for the presence of analytes in blanks.

1.9.2.2 Laboratory Control Sample

A laboratory control sample (LCS) is a sample of known composition that is spiked with all target analytes. The LCS is used with each analytical batch to determine whether the method is in control. Each analyte in the LCS shall be spiked at a level less than or equal to the midpoint of the calibration curve, which is defined as the median point of the curve instead of the middle of the range. The LCS shall be carried through the complete sample preparation and analysis procedure. The LCS cannot be used as the continuing calibration verification (CCV) sample.

At least one LCS shall be included in every analytical batch. If more than one LCS is analyzed in an analytical batch (e.g., LCS duplicate [LCSD]), results from all LCSs shall be reported. A failure of an analyte in any of the LCSs shall require appropriate corrective action, including qualification of the failed analyte in all of the samples, as required.

1.9.2.3 Surrogates

Surrogates are compounds used in organic compound analysis that are similar to the target analytes in chemical composition and behavior, but not normally found in environmental samples. Surrogates are used to evaluate accuracy, method performance, and extraction efficiency. Surrogates shall be added to environmental samples, controls, and blanks in accordance with the method requirements.

If a surrogate recovery is outside the acceptance limit, corrective action must be performed. After the system problems have been resolved and system control has been reestablished, the sample should be re-prepared and reanalyzed. If corrective actions are not performed or are ineffective, an appropriate flag shall be applied to the sample results.

1.9.2.4 Internal Standards

Internal standards (ISs) are known amounts of standards that are added to a portion of a sample or sample extract and carried through the entire procedure. ISs are used as a reference for calibration and for controlling the precision and bias of the analytical method. ISs shall be added to environmental samples, controls, and blanks, in accordance with the method requirements.

If the IS results are outside of the acceptance limits, corrective actions shall be performed. After the system problems have been resolved and system control has been reestablished, all samples analyzed

while the system was malfunctioning shall be reanalyzed. If corrective actions are not performed or are ineffective, an appropriate flag shall be applied to the sample results.

1.10 Project Assessment and Oversight

The QA programs are designed to measure performance and to monitor adherence to corporate QA policies, method, and project-specific requirements and regulatory requirements. Quality assessment is the responsibility of QA staff or designees, and is performed in cooperation with the affected operating group personnel. The purpose of this section is to establish standard procedures for quality assessment and the formulation of assessment criteria. Systems for corrective action are discussed in Section 1.9.3.

1.10.1 Project Audits/Assessments

The following project audits and assessments are planned:

- *Pre-mobilization audit (readiness review).* Prior to project mobilization, a systematic evaluation of readiness for field activities will be performed.
- *Well records review.* Once per well, a review of the documents and records associated with drilling activities will be reviewed. Verify accuracy and completeness. Verify that all planned activities were documented and that all associated documentation has been completed.
- *Subcontract laboratory sample receipt information.* Verify accuracy and completeness. Verify that discrepancies were documented and resolution of any potential data quality issues. Verify that all planned samples were collected and documented. Compare planned samples with the CoC forms and laboratory login information for samples actually submitted to the laboratory. Verify that corrective action is documented, if required and appropriate, for any data quality issues identified.

1.10.2 Audit Reporting

The response to audit or assessment findings may require written documentation, verbal communication, or formal corrective action. The timeframe and format of the response depends on the audit or assessment findings. Findings should include a discussion of the audit, explaining each finding, its significance, and whenever possible, its root cause. Deviations from plans, policies or SOP, if any, shall be discussed in terms of whether they were warranted by the circumstances of the project, and why. Finally, recommended corrective actions should be provided, if required and appropriate, as determined by the manager and technical personnel involved in the audit.

1.10.3 Corrective Action

Feedback and corrective action are required when deviations from documented policies, procedures or QC, if any, occur. These deviations may be identified through audits, data review and assessment, or staff feedback. The following sections describe the general procedures to be followed when departures from documented policies, procedures, or QC have occurred.

1.10.3.1 Audit Findings

The Shell Environmental Compliance Engineer and/or QA Auditor (member of the Shell Environmental / Science Department) shall be responsible for initiating and recommending corrective action, if any, in

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response to audit findings. Items requiring corrective action may require written documentation addressed to the Wells Superintendent. Corrective action steps shall be fully documented to assist in determining future corrective action procedures. When satisfactory progress has been achieved on each requested action, the Wells Superintendent or designee enters descriptions of actions and results on the form, retains a copy and returns the original to the Shell Environmental Compliance Engineer to close out the corrective action. The Shell Environmental Compliance Engineer maintains a file of Service Quality Non-Conformance Reports and keeps track of their progress. Unresolved corrective action requests, if any, are listed in a QA report to Management.

1.10.3.2 Data Quality Issues

Staff reviewing permit compliance data shall be responsible for requesting corrective actions in response to deficiencies that affect data quality and usability. To identify any potential impacts on the analytical results, a data usability summary shall include an evaluation of the data as applicable to the analytical method and GP requirements. Repetitive QC problems may result in staff retraining or subcontractor disqualification.

1.10.3.3 Staff Feedback

Each person involved in the data accumulation process shall be responsible for recognizing and reporting deviations, if any, from documented policies, procedures, and/or QC. If required and appropriate, deviations shall be documented and addressed to Shell. Analysts, Compliance Specialists, supervisors, and QC personnel shall use this system to document problems, deviations, and discrepancies encountered during sample collection, sample analysis, or data reporting, if any.

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2.0 Compliance Sampling Program for Operational Discharges

This section presents the applicable compliance requirements for operational discharges in the Authorization to Discharge under the NPDES GP for Oil and Gas Exploration Facilities on the OCS in the Chukchi Sea, Permit Number AKG-28-8100 issued by the USEPA, in compliance with the provisions of the Clean Water Act, 33 U.S.C. §1251 et seq., effective November 28, 2012.

2.1 Introduction and Overview

In the management of its activities, Shell has contracted with M-I SWACO for its compliance assurance for the exploration program in the Chukchi Sea on the *Polar Pioneer.* This section addresses those GP compliance tasks that will be performed by M-I SWACO under contract to the permittee during offshore drilling exploration. GP discharge limitations and monitoring requirements applicable to the offshore drilling exploration program in the Chukchi Sea are presented in Tables 2-1 through 2-5. The following sections detail the compliance monitoring requirements for each discharge.

Table 2-1 Effluent Limitations and Monitoring Requirements for Water-Based Drilling Fluids/Drill Cuttings (Discharge 001)

Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	Seasonal Restrictions
		Average Monthly Limit	Maximum Daily Limit		
Discharge 001 Water-Based Drilling Fluids and Drill Cuttings	SPP toxicity [note 1,10]	Minimum 96-hour LC_{50} of 30,000 ppm		Weekly Grab & End of Well [note 2]	Not Applicable
	Drilling fluids and cuttings	Static Sheen Test [note 3,10]		Daily Grab	
	Free oil	No Discharge [note 4,10]		Daily Grab	
	Diesel oil	No Discharge [note 5,10]		Daily Grab	
	Mercury	1 mg/kg [note 6]		Grab Once/well	
	Cadmium	3 mg/kg [note 6]		Grab Once/well	
	pH	Report (s.u.)		Grab Once/ well	
	Total Aqueous Hydrocarbons (TaqH)	Report (µg/L)		Grab Once /well [note 7]	
	Total Aromatic Hydrocarbon (TAH)	Report (µg/L)		Grab Once /well [note 8]	
	Total volume	Report (gal)		Daily Estimate [note 9]	

Notes:

LC_{50} = lethal concentration, 50% (in water, that kills 50% of the inhabitants in a given time)
mg/kg = milligram per kilogram
MLLW = Mean lower low water
µg/L = microgram per liter
ppm = parts per million
s.u. = standard units
SPP = suspended particulate phase

1. As determined by the 96-hour suspended particulate phase (SPP) toxicity test in accordance with Appendix 2 to Subpart A of 40 CFR Part 435, Drilling Fluids Toxicity Test. The discharge of water-based drilling fluids or drill cuttings generated using drilling fluids with a daily minimum or monthly average minimum 96-hour LC_{50} of less than 30,000 ppm is prohibited. If inclement weather conditions affect timely deliveries of samples, the permittee must notify EPA within 24 hours and document the conditions and rationale in the following monthly DMR.
2. See requirement of Section II.B.5.b. (Mineral Oil Pill). At the end-of-well, a sample must be collected for SPP toxicity testing where no mineral oil pill is used. The end-of-well sample can also serve as the monthly monitoring sample.
3. No discharge allowed upon failure of the static sheen test as determined in accordance with Appendix 1 to Subpart A of 40 CFR Part 435, Static Sheen Test.

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4. As determined by the static sheen test in accordance with Appendix 1 to Subpart A of 40 CFR Part 435, Static Sheen Test.

5. The discharge of drilling fluids or drill cuttings generated using drilling fluids which contain diesel oil is prohibited. Compliance will be demonstrated by gas chromatograph (GC) analysis of drilling fluid collected from the drilling fluid used at the greatest well depth ("end-of-well" sample) and of any drilling fluids or cuttings which fail the static sheen test compared to GC analysis of diesel oil in storage at the facility. Whenever drilling fluids or drill cuttings fail the static sheen test, the permittee is required to analyze an undiluted sample of the material which failed the test to determine the presence or absence of diesel oil in accordance with EPA SW846 Method 8015C (2007). Gas chromatography/mass spectrometry (GC/MS) may be used if an instance should arise where the permittee and the Director or DEC determine that greater resolution of the drilling fluid "fingerprint" is needed for a particular drilling fluid sample.

6. Dry weight in the stock barite. Results must be expressed as mg/kg (dry weight) of barite. The permittee must analyze a representative sample of stock barite once prior to drilling each well and submit the results with the DMR for the month in which drilling operations commence for the respective well. If any analytical result exceeds the mercury or cadmium effluent limitations in Table 1, the permittee must report the results to the Director in accordance with Section III.G., including the twenty-four hour notice of noncompliance requirement, of this GP. If the permittee uses the same supply of stock barite to drill subsequent wells, the permittee may submit the same analysis for those subsequent wells if no new supplies of barite have been received since the prior analysis. In this case, the DMR should state that no new barite was received since the last reported analysis.

7. As determined by summing the results of EPA Method 602 (plus Xylenes) or EPA Method 624 to quantify monoaromatic hydrocarbons and to measure TAH and EPA Method 610 or EPA Method 625 to quantify polynuclear aromatic hydrocarbons listed in EPA Method 610. Sample must be collected at the same time as the SPP toxicity test, to the extent practicable, and at the end of well.

8. As determined by EPA Method 602 (plus Xylenes) or EPA Method 624. Sample must be collected at the same time as the SPP toxicity test, to the extent practicable, and at the end of well.

9. Record separate total daily volumes of drilling fluids and drill cuttings and report the separate daily volumes in the End of Well Report. Report combined total volume of drilling fluids and drill cuttings discharged on a calendar day in the DMR.

10. The permittee must report the following discharge occurrences of noncompliance to the Director in accordance with Section III.G.l., including the twenty-four hour notice of noncompliance requirement, of this GP: (a) exceedance of the SPP toxicity limitation; (b) failure of the static sheen test; or (c) presence of diesel oil.

Table 2-2 Discharge Rate Limitations and Monitoring Frequency for Water-Based Drilling Fluids and Drill Cuttings (Discharge 001)

Water Depth [note 1]	Rate of Discharge [note 2]	Measurement Frequency	Sample Type
0 to 5 meters	No discharge	Hourly during discharge [note 3]	Estimate [note 4]
>5 to 20 meters	500 bbl/hr		
>20 to 40 meters	750 bbl/hr		
>40 meters	**1000 bbl/hr**		

Notes:

bbl/hr = barrels per hour

1. As measured from the MLLW.
2. Rate of discharge limitations do not include entrained seawater.
3. The maximum daily discharge limitation is calculated by multiplying the maximum hourly rate of discharge by 24 hours. For purposes of reporting, each hourly measurement must be recorded for each calendar day of discharge within the month. The monthly average limit is the average of the maximum daily hourly rate for each calendar day.
4. One-TRAX software based on operational data input and Pit Volume Totalizer.

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Table 2-3 Effluent Limitations and Monitoring Requirements for Deck Drainage (Discharge 002)

Discharge	Effluent Parameter	Effluent Limitations	Monitoring Requirements
Discharge 002 Deck Drainage	pH	s.u.	Monthly sample
	Free oil	No discharge [note 1]	Grab sample and visual per discharge event
	Total Volume (gal)	-	Monthly estimate
	TAqH	µg/L	Grab sample per discharge event [note 2,4]
	TAH	µg/L	Grab sample per discharge event [note 3,4]
	WET	TU_c	II.A.13.g.1.(page 22) and II.A.13.n.(page 26) of the NPDES GP [note 5]

Notes:

µg/L = microgram per liter

gal = gallons

s.u. = standard units

WET = whole effluent toxicity

1. Once per discharge event, the permittee must sample deck drainage discharges that are processed through an oil-water separator and test for sheen using the static sheen test in accordance with Appendix 1 to Subpart A of 40 CFR Part 435, Static Sheen Test. For discharges during unstable or broken ice conditions, a water temperature that approximates surface water temperatures after breakup must be used. During periods of discharge, the permittee must also conduct a visual observation for visual sheen as determined by the presence of a film or sheen upon or a discoloration of the surface of the receiving water.
2. As determined by summing the results of EPA Method 602 (plus Xylenes) or 624 to quantify monoaromatic hydrocarbons to measure TAH and EPA Method 610 or EPA Method 625 to quantify polynuclear aromatic hydrocarbons.
3. As determined by EPA Method 602 (plus Xylenes) or EPA Method 624.
4. Sample must be collected from the oil-water separator effluent.
5. Only applicable if initial toxicity screening exceeds threshold criteria outlined in Section 2.4.3.1. Also required once per well if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system.

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Table 2-4 Effluent Limitations and Monitoring Requirements for Sanitary and Domestic Wastes (Discharges 003 and 004)

Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	
		Average Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Discharge 003 Sanitary Waste	Flow (mgd)	----	----	Daily	Measured/Recorded
	BOD_5	30 mg/L	60 mg/L	Weekly	Grab or composite [note 1]
	TSS	30 mg/L	60 mg/L	Weekly	Grab or composite [note 1]
	Floating solids	No discharge		Daily	Visual [note 2]
	Foam	No discharge		Daily	Visual [note 2]
	Oily Sheen	No discharge		Daily	Visual [note 2]
	pH	6.5-8.5 s.u.		Weekly	Grab
	Fecal Coliform Bacteria	100 colonies/100 mL [note 3]	200 colonies/100 mL	Weekly	Grab
	Total Residual Chlorine [note 5]	----	1.0 mg/L	Weekly	Grab
	Flow	Report		Monthly	Meter
Discharge 004 Domestic Waste	pH	Report s.u.		Monthly	Grab
	Floating solids, garbage, foam	No discharge		Daily [note 2]	Visual
	Flow (mgd)	Report		Monthly	Estimated

Notes:

mgd = million gallons per day mg/L = milligram per liter mL = milliliters s.u. = standard unit

1. Composite samples may be collected in lieu of grab samples and must consist of at least four equal volume grab samples, two of which must be taken during periods of peak flow.
2. The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge and during conditions when observations on the surface of the receiving water are possible in the vicinity of the discharge. The observations and time of day must be recorded. The numbers of days floating solids, garbage, foam or oily sheen are observed must be recorded and reported in the DMR.
3. Must be reported as the geometric mean.
4. If inclement weather conditions affect timely deliveries of samples, the permittee must notify EPA within 24 hours document the conditions and rationale in the following monthly DMR.
5. Must be maintained as close to this concentration as possible. Sample must be collected immediately after chlorination and prior to any commingling of the waste streams. The analytical detection limit for this parameter is 0.1 mg/L. Residual chlorine may be monitored according to test procedures approved under 40 CFR Part 136 or using a Hach Test Kit capable of measuring free chlorine in the range of 0-3.5 mg/L with a sensitivity of 0.1 mg/L or better. Monitoring is not required if chlorine is not used as a disinfectant or for facilities serving fewer than 10 persons. One composite sample must consist of at least four equal volume grab samples, two of which must be taken during periods of peak flow.

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Table 2-5 Effluent Limitations and Monitoring Requirements for Miscellaneous Discharges

Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	
		Average Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Discharge 005 Desalination Unit Wastes	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1,2]		Once/discharge	Visual /Grab
	Total Volume	Report (gal)		Monthly	Meter
	WET	Report (TU_c)		Use rapid toxicity test 4X/well as initial screen.	Collect grab sample for analysis if results show potential toxicity or 1X/well if discharge >10,000 gal during 24 hr and if chemicals are added to the system.
Discharge 006 Blowout Preventer Fluid	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1,2]		Once/discharge	Visual /Grab
	Total Volume	Report (gal)		Monthly	Estimate
Discharge 007 Boiler Blowdown	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1,2]		Once/discharge	Visual /Grab
	Total Volume	Report (gal)		Monthly	Estimate
	WET	Report (TU_c)		Use rapid toxicity test 4X/well as initial screen.	Collect grab sample for analysis if results show potential toxicity or 1X/well if discharge >10,000 gal during 24 hr and if chemicals are added to the system.
Discharge 008 Fire Control System Test Water	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1,2]		Once/discharge	Visual /Grab
	Total Volume	Report (gal)		Monthly	Estimate
	WET	Report (TU_c)		Use rapid toxicity test 4X/well as initial screen.	Collect grab sample for analysis if results show potential toxicity or 1X/well if discharge >10,000 gal during 24 hr and if chemicals are added to the system.

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Discharge	Effluent Parameter	Effluent Limitations		Monitoring Requirements	
		Average Monthly Limit	Maximum Daily Limit	Sample Frequency	Sample Type
Discharge 009 Non-Contact Cooling Water	pH [note 3]	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1]		Daily	Visual
	Total Volume	Report (gal)		Daily [note 4]	Meter
	Temperature	Report (°F)		Continuous [note 4]	Measure
	WET	Report (TU_c)		Use rapid toxicity test 4X/well as initial screen.	Collect grab sample for analysis if results show potential toxicity or 1X/well if discharge >10,000 gal during 24 hr and if chemicals are added to the system.
Discharge 010 Uncontaminated Ballast Water	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1,2]		Once/discharge	Visual /Grab
	Total Volume	Report (gal)		Monthly	Estimate
Discharge 011 Bilge Water	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 5]		Once/discharge & Daily	Grab/Visual
	Total Volume	Report (gal)		Monthly	Meter
	WET	Report (TU_c)		Use rapid toxicity test 4X/well as initial screen.	Collect grab sample for analysis if results show potential toxicity or 1X/well if discharge >10,000 gal during 24 hr and if chemicals are added to the system.
Discharge 012 Excess Cement Slurry	pH	Report (s.u.)		Monthly	Grab
	Free oil	No discharge [note 1]		Once/discharge	Visual
	Total Volume	Report (gal)		Monthly	Estimate
Discharge 013 Mud, Cuttings & Cement at Sea Floor	Free oil	No discharge [note 1]		Daily	Visual
	Total Volume	Report (gal)		Monthly	Estimate

Notes:

°F = degrees Fahrenheit gal = gallons s.u. = standard units TU_c = chronic toxic units WET = whole effluent toxicity

1. Once per discharge event, the permittee must conduct a visual observation for visual sheen as determined by the presence of a film or sheen upon or a discoloration of the surface of the receiving water. The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge and

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during conditions when observations on the surface of the receiving water are possible in the vicinity of the discharge. The observations and time of day must be recorded. The number of days sheen is observed must be recorded and reported in the DMR. For discharges during unstable or broken ice conditions, a water temperature that approximates surface water temperatures after breakup must be used.

2. If visual observations of the discharge are not possible, the permittee must sample (grab sample) the discharge and test for sheen using the static sheen test in accordance with Appendix 1 to Subpart A of 40 CFR Part 435.
3. pH montoring and reporting is required. pH limit of 6.5-8.5 applies if chemicals are added to the system.
4. Estimated daily discharge volume and maximum and minimum recorded daily temperature must be reported for each outfall.
5. Once per discharge event, the permittee must sample bilge water discharges that are processed through an oil-water separator and test for sheen using the static sheen test in accordance with Appendix I to Subpart A of 40 CFR Part 435. For discharge during unstable or broken ice conditions, a water temperature that approximates surface water temperatures after breakup must be used. On a daily basis during discharge, the permittee must also conduct a visual observation for visual sheen as determined by the presence of a film or sheen upon or discoloration of the surface of the receiving water. The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge and during conditions when observations on the surface of the receiving water are possible in the vicinity of the discharge. The obesrvations and time of day must be recorded. The number of days sheen is observed must be recorded and reported in the DMR.

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2.2 Operational Discharge Compliance Program Roles and Responsibilities

Communication and coordination between compliance monitoring personnel, drilling operations management officials, and drillship operations officials will be critical to ensuring compliance with GP requirements. M-I SWACO NPDES Compliance Specialists must have a general understanding of oil and gas drilling procedures and vessel operations. They will be aware of the schedule for drilling operations and planned discharges of drilling fluids as well as discharges of materials related to vessel operations that are regulated under the GP.

The primary roles and responsibilities for individuals and teams responsible for implementing the operational discharge compliance program are defined in the following sections.

2.2.1 Wells Delivery Manager

The Wells Delivery Manager is the single individual responsible for the entire exploration program. In turn, the Wells Delivery Manager delegates authority to the Wells Superintendent, Engineering Team Lead and the Environmental Manager who are responsible for recognizing the requirements of this manual in their planning and budgeting and for implementing the QA program as assigned in this chapter. The Wells Delivery Manager has overall responsibility for the technical operation of the permit compliance program. Additional personnel will be assigned to assist in implementation of the quality program. The Wells Delivery Manager is specifically responsible for:

- Ensuring that personnel are free from any commercial, financial, or other undue pressures that may affect the quality of their work;
- Ensuring the implementation of the QA program as it pertains to operational discharge compliance under the GP;
- Ensuring that responsible, trained, and qualified personnel are assigned to manage, perform, or verify work affecting the quality of data; and
- Supervising personnel familiar with the procedures, the objective of the procedures, and the assessment of the results.

2.2.2 Wells Health, Safety and Environmental Department

The Wells Health, Safety and Environmental (HSE) Department has overall responsibility for the health and safety of employees as well as implementation of environmental regulatory programs. The HSE Department is specifically responsible for providing health and safety oversight in compliance with the requirements of the Shell HSE Program.

2.2.3 Shell Environmental Compliance Engineer

The Shell Environmental Compliance Engineer has independent reporting authority from the Wells Superintendent, Engineering Team Lead, and the Environmental Manager. The Environmental Compliance Engineer may assign personnel to assist in implementation of the quality program. The responsibilities of the Environmental Compliance Engineer are to:

- Conduct regularly scheduled audits of quality programs; and
- Advise operational managers of deficiencies (if any) in quality programs or performance.

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2.2.4 M-I SWACO NPDES Compliance Supervisor

The M-I SWACO NPDES Compliance Supervisor reports to the Wells Delivery Manager and is responsible for implementing the technical operation of the permit compliance program. The M-I SWACO NPDES Compliance Supervisor may assign personnel to assist with specific duties. Specific responsibilities of the M-I SWACO NPDES Compliance Supervisor include:

- Training M-I SWACO NPDES Compliance Specialists;
- Coordinating rig compliance laboratory assignments;
- Coordination with the Barrow mobile laboratory;
- Coordination with the fixed analytical laboratories;
- Providing overall direction to, and coordination of, quality programs as related to documenting compliance with the GP;
- Providing, and replacing as necessary, the equipment required for compliance tests;
- Approving and publishing written procedures;
- Reviewing Daily Activity Reports and associated records; and
- Archiving records at the end of drill season.

2.2.5 M-I SWACO NPDES Compliance Specialist

Personnel are responsible for complying with QA/QC requirements that pertain to their technical function. Each M-I SWACO NPDES Compliance Specialist shall have a combination of experience and education to adequately demonstrate a specific knowledge of their function and a general knowledge of operations, test methods, QA/QC procedures, and records management. M-I SWACO NPDES Compliance Specialists shall be responsible for recognizing and reporting deviations from documented policies, procedures, and/or QC to the M-I SWACO NPDES Compliance Supervisor or the Shell Environmental Compliance Engineer.

The responsibility for implementing the QA program ultimately rests with the M-I SWACO NPDES Compliance Specialist. The M-I SWACO NPDES Compliance Specialist is specifically responsible for:

- Maintaining technical proficiency in the compliance procedures;
- Maintaining records for field equipment;
- Maintaining and calibrating equipment for compliance tests;
- Collecting and/or analyzing effluent samples;
- Transferring documents to the records custodian at the end of the drilling season;
- Providing copies of analytical reports to the permittee; and
- Reporting deviations from procedures to supervisory personnel.

2.3 Data Quality Objectives

The DQOs for the monitoring of operational discharges are to address the following GP requirements:

- Ensure that samples and measurements taken for the purpose of monitoring are representative of the monitored activity;
- Collect and analyze effluent samples in accordance with the methods specified in 40 CFR Part 435 and 40 CFR Part 136; and
- Collect and analyze samples of the type, frequency, quantity, and quality to meet the effluent limitations and monitoring requirements outlined in Section II of the GP.

2.4 Field Sampling

The following sections outline the operational discharge compliance sampling program design, which is intended to meet project DQOs and the effluent limitations and monitoring required by the GP.

2.4.1 Sampling, Measurement, and Observation Locations

This section presents discharge sampling, flow/volume measurement, and observation locations. Samples of treated discharge media will be collected after the final treatment system component and prior to the applicable discharge point or caisson. Discharge flow rates will either be estimated (total volume discharged) or recorded by using a dedicated inline flow meter in accordance with M-I SWACO written procedures. Discharges that include a chemical additive and exceed 10,000 gallons during any 24-hour period will be sampled once per well for whole effluent toxicity testing in accordance with M-I SWACO written procedures. Visual discharge observations for sheen, floating solids, or other residues will be conducted from vantage points aboard the surface of the vessel that allow for unobstructed observation of the receiving waters in accordance with M-I SWACO SOP 3005.

Table 2-6 presents the discharge sampling locations, flow/volume measurement locations, and visual observation locations as well as station identifiers and vessel sample location designators. Vessel sample locations correlate to points identified on the *Polar Pioneer* in Figure 1 (Appendix B to the BMP).

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Table 2-6 Discharge Sampling Locations

Discharge	Location/ Station ID[1]	Sample Location(s)	Type of Measurement	Effluent Parameter
Discharges D001/D009/D012 Inboard Comingled Drilling Fluids and Cuttings, Non-Contact Cooling Water, and Excess Cement Slurry	Lower Hull Plan Figure Shunt line at Port #2 Column	Drilling fluids and cuttings from shale shakers	Analytical sample	SPP toxicity
				TAH
				TAqH
				Diesel oil
			Field sample	pH, Static sheen
			Estimate	Flow Volume (mgd)
			Visual observation	Free Oil (visual sheen)
Discharges D002/D005/D009/D011 Deck Drainage, Desalination Unit Wastes, Non-Contact Cooling Water, Bilge Water	Lower Hull Plan Figure GSSL discharge	OWS Effluent line; Flow meter on OWS	Analytical sample	TAH
				TAqH
				Tox screen/WET
			Field sample	pH
			Calculation/Measurement	Total Volume (gal)
			Visual observation	Free Oil (visual sheen)
Discharge D003 Sanitary Wastewater	Lower Hull Plan Figure	MSD Effluent Line	Analytical sample	BOD_5
				TSS
				Fecal Coliform Bacteria
			Field sample	pH, Total residual chlorine
			Flow meter reading	Flow Volume (mgd)
			Visual observation	Oily Sheen, Floating solids & garbage, Foam
Discharge D004 Domestic Wastewater	Lower Hull Plan Figure	Effluent Line	Field Sample	pH
			Estimated	Flow Volume (mgd)
			Visual Observation	Floating solids & garbage, Foam
Discharge 010 Uncontaminated Ballast Water	Lower Hull Plan Figure	Inboard Ballast Water Discharge Lines	Field sample	pH
			Calculation/Measurement	Total Volume (gal)
			Visual observation	Free Oil (visual sheen)

Notes:

[1] Locations are identified in Discharge Locations Figures, Appendix B to the BMP.

bbl/hr = barrels per hour
BOD_5 = 5-day biochemical oxygen demand
BOP = blowout preventer
gal = gallons
GSSL = General Seawater Service Line
mgd = millions of gallons per day
MSD = marine sanitation device
OWS = oil/water separator
SPP = suspended particulate phase
TSS = total suspended solids
WET = whole effluent toxicity

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2.4.2 Sampling, Measurement, and Observation Frequencies

This section summarizes the procedures for ensuring compliance with the required sampling, measurement, and observation frequencies specified in the GP. Frequencies of general tasks related to the operational discharge compliance sampling program are summarized in Table 2-7.

Compliance monitoring requirements and sampling tasks for each discharge are presented in the following sections. All sampling related activities shall be conducted in accordance with this QAPP and the applicable written procedures.

2.4.2.1 Daily Tasks

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Attend project health and safety meetings and project planning meetings and provide input and communication as it relates to sampling requirements;
- Maintain and document the condition of the sample storage refrigerator in accordance with the procedures outlined in this QAPP and M-I SWACO SOPs 1005 and 1006;
- Provide Daily Activities Report to the M-I SWACO NPDES Compliance Supervisor;
- Document flow/volume estimates and sampling /testing performed to ensure compliance with M-I SWACO written procedures and permit stipulations;
- Monitor to ensure if a chemical additive is used and if discharges from 002 (deck drainage), 005 (desalination unit wastes), 008 (fire control system test water), 009 (non-contract cooling water) and 011 (bilge water) exceed 10,000 gallons during any 24-hour period;
- Collect samples and submit to a contract laboratory (Environ or equivalent laboratory). Perform initial toxicity screening four times per well in accordance with the M-I SWACO SOP and the analytical protocols established by Environ for conducting the required toxicity testing. WET testing required if any initial toxicity screening does not meet criteria listed in Section 2.4.3.1.

2.4.2.2 Weekly Tasks

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a weekly basis:

- Inspect and document the current inventory of sampling equipment, supplies, and containers in accordance procedures outlined in this QAPP and M-I SWACO SOP 1006;
- Request additional materials as necessary; and
- Document weekly measurements and sampling /testing performed to ensure compliance with M-I SWACO written procedures and permit stipulations.

2.4.2.3 Monthly Tasks

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Document or estimate monthly discharge flow volumes from daily measurements in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006 for all discharges; and
- Provide a monthly discharge flow volume report to the Shell Environmental Compliance Engineer and M-I SWACO NPDES Compliance Supervisor detailing all discharges performed during the month, including daily discharge volume measurements.

Table 2-7 Summary of Compliance Sampling, Measurement, and Observation Frequencies and Related SOPs

Frequency	Task	M-I SWACO SOP
Daily	Attend HSE and project planning meetings	N/A
	Maintain sample refrigerator	1005, 1006
	Daily activity report	N/A
	Monitor if chemicals are added to the system and if discharges exceed 10,000 gals/24-hr period	1006, 2001, Section 2.4.3
Weekly	Sampling supply inventory	1006
Monthly	Document or estimate monthly discharge flow volumes	1006
	Monthly discharge flow volume report	N/A

Notes:

HSE = Health, Safety, and Environment
SOP = standard operating procedure

2.4.3 Compliance Sampling Requirements for Each Operational Discharge

Compliance activities include the collection of samples for analysis at the rig compliance laboratory, and collection of samples for shipment to the Barrow mobile laboratory and fixed subcontract laboratory. Environmental samples will be collected using methods selected to ensure that project DQOs are met. Written procedures that outline the procedures to be followed for the collection and analysis of environmental samples are included as an attachment to this QAPP. These written procedures ensure that project personnel collect representative samples in a consistent manner for all required sampling matrices and locations, that contamination is not introduced during collection, and that the sample volumes are properly preserved in order to meet project objectives and analytical method requirements.

The following sections describe the samples to be collected for each discharge to support compliance with this QAPP and the GP. Several discharges are comingled and will be sampled according to the most stringent effluent limitations among the individual discharges (Table 2-9). If necessary, individual discharges will be sampled upstream from the discharge point. Therefore, the following information is presented for the individual discharges: sampling tasks and frequency; the type and matrix of samples to be collected; analytical methods; required volume of sample; QC samples and frequency; and sample containers, preservation, and holding times.

2.4.3.1 Effluent Toxicity Testing

As part of the Phase II Assessment of the EMP, initial toxicity testing will be conducted four times per well during exploration drilling activities for the following discharges: 002 (deck drainage), 005

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(desalination unit wastes), 007 (boiler blowdown), 008 (fire control system test water), 009 (non-contact cooling water), and 011 (bilge water).

The effluent samples will be collected from the discharge stream after the last treatment on the drilling rig and before the discharge stream enters the receiving waters. If samples are collected for both toxicity testing and operational discharge compliance analyses at the same time, a split sample will be collected. Split samples are two or more representative portions taken from one sample collected at the appropriate sampling location. To ensure that split samples are representative, the volume of sample collected must equal the total volume required for all of the laboratory analyses. For example, approximately 51 L of effluent must be collected and split for a discharge of deck drainage that requires initial toxicity screening (2L), WET testing (45 L), TAH (0.12 L), TAqH (2 L), static sheen (0.5 L), and pH (0.1 L) analyses.

Effluent toxicity testing as required under the GP will consist of an initial toxicity screening with 100% effluent at four different time periods selected to reflect discharge practices and operational processes. If the initial toxicity screen indicates the effluent sample may cause adverse biological impacts as defined by the toxicity testing threshold limits established for this program, then whole effluent toxicity (WET) testing is required and the WET testing sample will be collected as soon as practical. In addition, if other discharge thresholds are exceeded as specified greater than 10,000 gallons in a 24 hour period and if chemicals are added such that they will end up in the discharge, then WET testing will also be initiated.

The threshold limits established for this program are based on the initial toxicity screening test using echinoderm fertilization success. This test has been demonstrated to have a detectable significant criteria ranging (i.e., ability to detect and effect) from 15.5% to 25% for various laboratories (Carr et al. 1996, USGS 1998, and Porebski et al. 1999). For this program, the initial toxicity screening thresholds include the following three criteria, which must all be met to indicate a positive toxicity result:

1) Test validation requires >70% fertilization.
2) A sample that has sufficient reduction in fertilization to be identified as an adverse effect requires a statistically significant difference between the validated control fertilization test and the 100% effluent.
3) The decline in fertilization compared to the validated control response must be at least 25%.

For example, if the control percent fertilization was 80%, then the effluent response *must* be statistically significantly different from the control and have exhibited a greater than 25% difference in percent fertilization.

The remote location of the drilling sites in the Chukchi Sea creates logistical challenges for transporting samples to the laboratory within the method-required holding times. Due to these challenges, the volume of effluent initially collected to conduct the WET testing will include the required renewal water (if WET testing is triggered). An adequate volume for each effluent sample will be collected to conduct the rapid screening test and WET testing, if triggered (2 L for rapid screen test and 45 L for three WET 7-day chronic assays), for a particular discharge, including renewal water volume, unless the discharge is intermittent. For discharges with an intermittent discharge stream, the rapid screening test and the WET testing sample (if triggered) will be collected at different time points. The practice of collecting all volume required for the WET testing requirements up front has been used to assess the chronic toxicity of

discrete discharge events such as storm water runoff or de-icing activities, where renewal samples are not able to be collected beyond the time period of the event in question. Effluent samples will be collected for toxicity testing in accordance with the Environ *Collection of Effluent Samples for Biological Testing Memorandum* dated 20 May 2013 (Appendix A).

Testing should be initiated on samples within 36 hours of sample collection, but must not exceed 72 hours as prescribed in the GP and method guidance. Effluent samples used for test solution renewals on the WET 7-day chronic tests (days 1-6) may be used up to 48-hours after the initial use (test initiation) (USEPA 2002). This guidance indicates that samples may be used for test solution renewals at up to 120 hours from the time of collection. Table 2-8 summarizes these holding times.

Table 2-8 Effluent Sample Holding Times for Toxicity Testing

Holding Time for Test Initiation	Sample Aging for Use as Test Solution Renewal (During Test)
0 – 36 hours (recommended) 0 – 72 hours (allowed maximum)	0 – 84 hours (recommended) 0 – 120 hours (allowed maximum)

Note:
Holding times are from time of sample collection.

All toxicity testing will be conducted at the Environ Port Gamble Environmental Laboratory located in Port Gamble, Washington. In order to meet holding times for testing, effluent sample collection for an applicable discharge event will be coordinated with helicopter transport and priority air freight shipping from Barrow, Alaska.

An example scenario and timeline for effluent sample collection and shipment is provided below. The timeline allows for minor delays in transport. Alaska Airlines has ~20 flights each day from Anchorage to Seattle. Departure of samples from Anchorage can occur aboard an earlier or slightly later flight to meet the 36 hour holding time (initiate test by 2000 on second day).

0800-1200 Effluent samples are collected during the morning of the applicable discharge event.

1200-1300 All samples are packed with ice packs in coolers and CoC procedures are initiated as described in this QAPP.

1300-1400 Samples are transported in the afternoon by helicopter to Alaska Air Cargo GoldStreak® in Barrow, Alaska.

1843-2035 Samples are transported by cargo only or a cargo/passenger aircraft (cargo and passengers) in the early evening to Anchorage, Alaska.

0150-0615 Samples are transported by cargo only, combination of cargo and passenger, or passenger aircraft in the early morning to Seattle, Washington. Alaska Air Cargo GoldStreak® known shipper status allows transport on passenger aircraft for packages less than 100 pounds.

0900-1200 Samples are transported by courier to Environ Port Gamble Environmental Laboratory in the morning for sample log-in and test initiation by the afternoon.

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If WET testing is required, 45 L will be collected at the time of initial sample collection. This initial volume of sample for WET testing, which includes renewal water volume, will be assigned Priority 1 (Type I Samples) in the helicopter support priority matrix due to the short holding time for test initiation (Table 2-8). Subsequent 15-L volumes of renewal water will be collected as required by holding time remaining on the initial aliquot and compliance with the analytical method, and an attempt to transport this renewal water will be made. Transportation of renewal water samples will be assigned transport Priority 3 (Crew Change) priority.

If transport of the renewal water sample is delayed, the laboratory will use the initial aliquot that was received; M-I SWACO NPDES Compliance Specialists will continue to attempt to collect the required volume of renewal water during the next feasible time.

The laboratory will document the condition of the sample volume used for WET testing, including any variance in receipt of the sample (such as weather delays, or delays due to competing helicopter priorities); the nature of the waste stream and effect on toxicity degradation (if any); and the action taken by the laboratory (e.g., utilization of the initial sample). Shell will notify EPA of any variance in samples collected for WET testing.

2.4.3.2 Compliance Sampling for Water-Based Drilling Fluids and Drill Cuttings (Discharge 001)

Prior to drilling each well, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks:

- Stock barite is analyzed and assigned a lot number by the vendor and a Certificate of Analysis accompanies each shipment. The M-I SWACO NPDES Compliance Specialist will retain a copy of the Certificate of Analysis in the field notebook for the lot of stock barite prior to its use. Each time a new lot batch of barite is utilized within the drilling season, the M-I SWACO NPDES Compliance Specialist will retain the Certificate of Analysis and record the lot number in the field notebook prior to its use.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks while discharging water-based drilling fluids and cuttings:

- Document the flow volume from discharge flow meters or estimate the volume of fluids and cuttings discharged hourly by calculating the hourly discharge rate in bbl/hr, and ensure that the depth-dependent discharge rate in Table 2-2 is not exceeded. The Shell Environmental Compliance Engineer and M-I SWACO NPDES Compliance Supervisor will be notified when the discharge rate exceeds 75% of the maximum allowable hourly discharge rate and there is potential for that rate to be exceeded.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks on a daily basis:

- Document the flow volumes from all discharge flow meters and waste volumes in bulk discharge holding tanks aboard the vessel in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006. The total daily volumes of drilling fluids and drill cuttings will be recorded

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and reported in the End of Well Report, and total volume discharge per calendar day will be recorded daily and reported monthly in the Discharge Monitoring Report (DMR).

- Perform and document visible sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- Collect samples and document results for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004. As soon as practicable, perform static sheen analysis.
- Document the quantity of any chemical additive used.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks on a weekly basis:

- Collect and document a water-based drilling fluids and cuttings sample SPP toxicity in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, and 2002. Immediately transfer the SPP toxicity sample to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003. Retain one SPP toxicity sample container in the sample refrigerator until acceptable results are received for the sample transported to the fixed analytical laboratory. If problems occur with the original SPP toxicity sample transported to the analytical laboratory that will invalidate the results, package and transport the remaining sample to the fixed analytical laboratory for analysis.

Once per well, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. Immediately deliver pH samples to the rig compliance laboratory for analysis.
- Collect and document a water-based drilling fluid sample for TAH and TAqH in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, and 2008. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003. Samples must be collected at the same time as the SPP toxicity sample, to the extent practical (see end of well tasks below).
- When total depth of the well (end of well) has been reached or static sheen test fails, the M-I SWACO NPDES Compliance Specialist will collect and document a water-based drilling fluid sample for diesel oil (fingerprint) analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, and 2008. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

- When total depth of the well (end of well) has been reached, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks: Collect and document a water-based drilling fluids and cuttings sample for SPP toxicity in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, and 2002. Immediately transfer the sample to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003. Retain one SPP toxicity sample container in the sample refrigerator until acceptable results are received for the sample transported to the fixed analytical laboratory. If problems occur with the original SPP toxicity sample transported to the fixed analytical laboratory that will invalidate the results, package and transport the remaining sample to the fixed analytical laboratory for analysis.

- In support of the EMP, two samples of water-based drilling fluids and two samples of drill cuttings will be collected during drilling of the largest casing interval (after the blowout preventer (BOP) is set), and two samples of used water-based drilling fluids will be collected during bulk-mud discharge (if applicable), in Phase II of the monitoring program for a total of six samples (Table 3-4). Drilling-mud compositions and monitoring records will be obtained from Transocean to the degree possible. The drilling phases targeted for this sampling include 1.) drilling the largest casing interval after the BOP is set; and 2.) bulk-mud discharges (if this occurs). The water-based drilling fluids and drill cuttings will be collected in clean glass jars, and sample handling and analysis will be performed in accordance with Section 3.0. Samples will be analyzed for the metals identified in Table A of the GP and for hydrocarbons (specific suite of analytes and individual analytes reported in Section 3.0).

- As required by Section II.A.13.j.1 of the GP, samples will be collected for the analysis of each drilling fluids system for the metals identified in Table A of the GP. A sample will be collected from each drilling fluids system at the three casing intervals of the lower hole section as described in the Drilling Fluid Plan and in accordance with Section 3.4.1.5. Samples of the drilling fluids system must be collected when the metals concentrations are projected to be at their maximum value for each applicable system. Samples of water-based drilling fluids will be collected in clean glass jars, and sample handling and analysis will be performed in accordance with Section 3.0.

Table 2-9 summarizes the drilling fluid and effluent monitoring tasks and frequencies, and Table 2-10 summarizes the analytical samples that will be collected for Discharge 001, Water-Based Drilling Fluids and Drill Cuttings.

Table 2-9 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 001

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
	Visual sheen tests	1006, 3005
	Static sheen tests	1006, 2001, 3004
Prior to drilling	Stock barite metals Certificate of Analysis	1006

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Frequency	Task	M-I SWACO SOP
Weekly	SPP toxicity sample	1006, 2001, 2002, 2003, Section 2.4.3
Once per well	pH samples of drilling fluid	1006, 2001, 2012
	TAH/TAqH samples of drilling fluid	1006, 2001, 2003, 2008
End of well	SPP toxicity sample	1006, 2001, 2002, 2003, Section 2.4.3
	Diesel fingerprint sample of aqueous drilling fluid	1006, 2001, 2003, 2008
During discharge	Daily volume measurements and flow rate estimates for discharge of water-based drilling fluids/drill cuttings	1006

Notes:

HSE = Health, Safety, and Environment
SOP = standard operating procedure
SPP = suspended particulate phase
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons

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Table 2-10 Analytical Sampling for Water-Based Drilling Fluids and Drill Cuttings (Discharge 001)

Type of sample	Matrix	Parameter	Analytical Method	Permit Limit/ Water Quality Standard	Required Volume	Sample Container	Preservative	Holding Time	Sampling Frequency
Mud & cuttings from shale shakers	Aqueous	Suspended particulate phase (SPP) toxicity	40 CFR 435, App. 2 to Subpart A (E1619)	96-hr LC_{50} > 30,000 ppm	1 L	(1) 1-gal (4L) LDPE jar	0 to 4°C	90 days	Weekly and End of well
		TAH/TAqH[1]	E624	Report (µg/L)	40 mL	(3) 40-mL VOA vials w/TLS	Cool <6°C, HCl to pH<2	14 days	Once per well
			E625 SIM	Report (µg/L)	1 L	(2) 1-L amber glass jars w/TLC	Cool <6°C	7 days to extraction, 40 days to analysis	
	Aqueous	Diesel fingerprint	SW8015C	No discharge	10 mL	(2) 40-mL VOA vials w/TLS	Cool <6°C	7 days to extraction, 40 days to analysis	End of well, or if static sheen test fails
	Oil				10 mL	(2) 40-mL VOA vials w/TLS			

Notes:

All samples will be collected as grab samples.

[1] Sample must be collected at the same time as the SPP toxicity test, to the extent practicable, and at the end of well.

µg/L = micrograms per liter
mg/kg = milligrams per kilogram
mL = milliliter
L = liter
HCl = hydrochloric acid
LDPE = low-density polyethylene
OWS = oil/water separator
SPP = suspended particulate phase
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons
TLC = Teflon-lined cap
TLS = Teflon-lined septa
VOA = volatile organic analysis

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2.4.3.3 Compliance Sampling for Deck Drainage (Discharge 002)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect samples and document samples for pH analysis in accordance with QAPP procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, transport pH samples to the rig compliance laboratory for analysis.

The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well:

- Four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3 of this QAPP.
- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document three samples from the OWS effluent on an every-other-day basis in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks once per discharge event (if conducted continuously):

- Collect and document samples from the OWS effluent for TAH and TAqH in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, and 2008. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.
- Document the quantity of any chemical additive used.
- Perform and document visible sheen test in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- Document the flow volumes from the OWS effluent flow meter in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006. Additionally, the volume discharged directly overboard will be estimated and recorded. The total volume in gallons will be reported monthly.
- Collect samples for static sheen tests from OWS effluent and document results in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004. Immediately deliver samples to the rig compliance laboratory for analysis.

Table 2-11 summarizes the effluent monitoring tasks and frequencies, and Table 2-12 summarizes the analytical samples that will be collected for Discharge 002, Deck Drainage.

Table 2-11 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 002

Frequency	Task	M-I SWACO (or Misc.) SOP
Once per discharge event (if conducted continuously)	Document discharge flow volumes	1006
	TAH/TAqH samples of deck drainage	1006, 2001, 2003, 2008
	Visual sheen test	1006, 3005
	Static sheen test	1006, 2001, 3004
Monthly	pH sample	1006, 2001, 2012
Four times per well	Initial effluent toxicity screening	1006, 2001, Environ001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06

Notes:

SOP = standard operating procedure
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons
WET = whole effluent toxicity

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Table 2-12 Analytical Sampling for Deck Drainage (Discharge 002)

Type of sample	Matrix	Parameter	Analytical Method	Effluent Limit/ Water Quality Standard	Required Volume	Sample Container	Preservative	Holding Time	Sampling Frequency
OWS Effluent	Aqueous	TAH/TAqH	E624	Report (µg/L)	40 mL	(3) 40-mL VOA vials w/TLS	Cool <6°C, HCl to pH<2	14 days	Once per discharge event
			E625 SIM	Report (µg/L)	1 L	(2) 1-L amber glass jars w/TLC	Cool <6°C	7 days to extraction,40 days to analysis	
		Initial toxicity screening	EPA/600/R-95-136 (TOX045A.01)	Report – Test is statistically significantly different from control AND there is >25% decline in fertilization relative to control	1-L	(1) 1-L LDPE bottle	0 to 4°C	90 days	Four times per well
		WET[2]	EPA/600/R-95-136 (Topsmelt Chronic 7d Survival and Growth Test)[1] or EPA/821-R-02-014 (Menidia Chronic 7d Survival and Growth Test)[1]	Report	30-L	(3) 10-L LDPE cubitainers	Cool <6°C	36 hours (Max 72 hrs) and requiring justification for holding time if >36 hour)	Upon failure of toxicity screening criteria, OR Once per well if discharge exceeds 10,000 gal and chemicals are added to the system
			EPA/821-R-02-014 (Mysid Chronic 7d Survival, Growth, and Fecundity Test)[1]		30-L	(3) 10-L LDPE cubitainers			

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Type of sample	Matrix	Parameter	Analytical Method	Effluent Limit/ Water Quality Standard	Required Volume	Sample Container	Preservative	Holding Time	Sampling Frequency
OWS Effluent	Aqueous	WET[2]	EPA/600/R-95-136 (Echinoderm Larval Development Test)		1-L	(1) 1-L LDPE bottle			

Notes:

All samples collected as grab samples.

[1] Samples for these tests should be provided as one, 10-L sample on an every-other-day basis. If this sample strategy is not feasible, a single sample of 30 L may be provided at one time.

[2] Samples for WET testing may be collected in larger containers (up to 20-L LDPE cubitainers or different combination thereof) to facilitate sufficient volume to conduct all three tests.

µg/L = micrograms per liter
mL = milliliter
L = liter
EPA = U.S. Environmental Protection Agency
HCl = hydrochloric acid
LDPE = low-density polyethylene
OWS = oil/water separator
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons
TLC = Teflon-lined cap
TLS = Teflon-lined septa
VOA = volatile organic analysis
WET = whole effluent toxicity

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2.4.3.4 Compliance Sampling for Sanitary Wastes (Discharge 003)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the sanitary waste flow volume from the effluent flow meter in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.
- Perform and document visual assessments for floating solids, sheen, debris, sludge, foam, deposits, scum, or other residues from discharges of sanitary wastes in accordance with QAPP Section 3.0 and M-I SWACO SOP 1006.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a weekly basis:

- Collect and document treated sanitary waste samples for pH, BOD_5, TSS, fecal coliform (FC) bacteria, and total residual chlorine (if used as disinfectant) in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006, 2001, 2010, 2011, 2012, 2013, and 2014. Immediately deliver pH and total residual chlorine samples to the rig compliance laboratory for analysis. Immediately transfer BOD_5, TSS, and FC samples to the sample refrigerator for storage awaiting packaging for transportation to the Barrow mobile laboratory. Package samples for transport to the Barrow mobile laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

Table 2-13 summarizes the effluent monitoring tasks and frequencies, and Table 2-14 summarizes the analytical samples that will be collected for Discharge 003, Sanitary Wastes.

Table 2-13 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 003

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
	Visual assessments of receiving waters	1006, 3005
	Visual sheen tests	1006, 3005
Weekly	pH sample	1006, 2001, 2012
	BOD_5, TSS, Total residual chlorine samples	1006, 2001, 2003, 2010, 2013, 2014
	Fecal coliform bacteria sample	1006, 2001, 2003, 2011

Notes:
BOD_5 = 5-day biochemical oxygen demand
SOP = standard operating procedure
TSS = total suspended solids
WET = whole effluent toxicity

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Table 2-14 Analytical Sampling for Sanitary Wastes (Discharge 003)

Type of sample	Matrix	Parameter	Preparation and Analysis Method	Effluent Limit/ Water Quality Standard	Required Volume	Sample Container	Preservative	Holding Time	Sampling Frequency
Treatment System Effluent	Aqueous	BOD_5	SM5210-B	30 mg/L (Monthly average); 60 mg/L (Daily limit)	1 L	(1) 1-L poly bottle	Cool <6°C	48 hours	Weekly
		TSS	SM2540-D	30 mg/L (Monthly average); 60 mg/L (Daily limit)	1 L	(1) 1-L poly bottle	Cool <6°C	7 days	
		Fecal coliform bacteria	SM9222-D	100 colonies/100 mL (Monthly average); 200 colonies/100 mL (Daily Limit)	100 mL	(1) 125-mL sterile poly container w/ $Na_2S_2O_3$	Cool <6°C, Na_2SO_3	8 hours	

Notes:

All samples will be collected as grab samples.
L = liter
mg/L = milligrams per liter
mL = milliliter
BOD_5 = 5-day biochemical oxygen demand
$Na_2S_2O_3$ = sodium thiosulfate
TSS = total suspended solids

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2.4.3.5 Compliance Sampling for Domestic Wastes (Discharge 004)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the domestic waste flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.
- Perform and document visual assessments for floating solids, garbage, or foam from discharges of domestic wastes in accordance with QAPP Section 3.0 and M-I SWACO SOP 1006.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. Immediately deliver pH samples to the rig compliance laboratory for analysis.

Table 2-15 summarizes the effluent monitoring tasks and frequencies for Discharge 004, Domestic Wastes.

Table 2-15 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 004

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
	Visual assessments of receiving waters	1006, 3005
Weekly	pH sample	1006, 2001, 2012

Notes:
SOP = standard operating procedure

2.4.3.6 Compliance Sampling for Desalination Unit Wastes (Discharge 005)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.

The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well:

- Four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document

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initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP.

- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- Document the quantity of any chemical additive used.
- If visual sheen tests cannot be performed, collect and document samples for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-16 summarizes the effluent monitoring tasks and frequencies for Discharge 005, Desalination Unit Wastes.

Table 2-16 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 005

Frequency	Task	M-I SWACO (or Misc.) SOP
Daily	Document discharge flows and waste volumes	1006
Four times per well	Initial effluent toxicity screening	1006, 2001, ENV001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 2003, 2008
	Static sheen test (if required)	1006, 2001, 3004

Notes:
SOP = standard operating procedure
WET = whole effluent toxicity

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2.4.3.7 Compliance Sampling for Blowout Preventer Fluid (Discharge 006)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- If visual sheen tests cannot be performed, collect and document samples for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-17 summarizes the effluent monitoring tasks and frequencies for Discharge 006, Blowout Preventer Fluid.

Table 2-17 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 006

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 2003, 2008
	Static sheen test (if required)	1006, 2001, 3004

Note:
SOP = standard operating procedure

2.4.3.8 Compliance Sampling for Boiler Blowdown (Discharge 007)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.

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The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well:

- If possible four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Due to the infrequency of discharge and limited volume of water produced, it is unlikely that this screening will be able to be performed four times per well.
- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- If visual sheen tests cannot be performed, collect and document samples for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-18 summarizes the effluent monitoring tasks and frequencies for Discharge 007, Boiler Blowdown.

Table 2-18 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 007

Frequency	Task	M-I SWACO (or Misc.) SOP
Daily	Document discharge flows and waste volumes	1006
Four times per well	Initial effluent toxicity screening	1006, 2001, ENV001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, TOX002.05, TOX012.06, TOX014B.02, TOX043.06

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Frequency	Task	M-I SWACO (or Misc.) SOP
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 2003, 2008
	Static sheen test (if required)	1006, 2001, 3004

Notes:

SOP = standard operating procedure
WET = whole effluent toxicity

2.4.3.9 Compliance Sampling for Fire Control System Test Water (Discharge 008)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Estimate flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.

The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well:

- Four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP.
- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- If visual sheen tests cannot be performed, collect and document samples for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

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- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. Immediately deliver pH samples to the rig compliance laboratory for analysis.

Table 2-19 summarizes the effluent monitoring tasks and frequencies for Discharge 008, Fire Control System Test Water.

Table 2-19 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 008

Frequency	Task	M-I SWACO (or Misc.) SOP
Daily	Document discharge flows and waste volumes	1006
Four times per well	Initial effluent toxicity screening	1006, 2001, ENV001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 2003, 2008
	Static sheen test (if required)	1006, 2001, 3004

Notes:
SOP = standard operating procedure
WET = whole effluent toxicity

2.4.3.10 Compliance Sampling for Non-contact Cooling Water (Discharge 009)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis for each of the non-contact cooling water discharges:

- Document the flow volume from the effluent flow meters in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Perform and document visual sheen tests for each outfall in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- Temperature will be monitored continuously and documented for non-contact cooling water (009) in accordance with procedures outlined in this QAPP and M-I SWACO LWI-001.
- Document the quantity of any chemical additive used.

The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well*:*

- Four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document

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initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP.

- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-20 summarizes the effluent monitoring tasks and frequencies for Discharge 009, Non-contact Cooling Water.

Table 2-20 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 009

Frequency	Task	M-I SWACO (or Misc.) SOP
Daily	Document discharge flows and waste volumes	1006
	Visual sheen tests	1006, 3005
	Temperature monitoring of non-contact cooling water	1006, LWI-001
Four times per well	Initial effluent toxicity screening	1006, 2001, ENV001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Monthly	pH sample	1006, 2001, 2012

Notes:
SOP = standard operating procedure
WET = whole effluent toxicity

2.4.3.11 Compliance Sampling for Uncontaminated Ballast Water (Discharge 010)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.

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During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.
- If visual sheen tests cannot be performed, collect and document samples for static sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-21 summarizes the effluent monitoring tasks and frequencies for Discharge 010, Uncontaminated Ballast Water.

Table 2-21 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 010

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 2003, 2008
	Static sheen test (if required)	1006, 2001, 3004

Notes:
SOP = standard operating procedure
WET = whole effluent toxicity

2.4.3.12 Compliance Sampling for Bilge Water (Discharge 011)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the flow volume for discharges from the effluent flow meters in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive.

The M-I SWACO NPDES Compliance Specialist is responsible for performing the following sampling tasks four times per well:

- Four times per well, at intervals designated to be representative of the discharge's toxicity, a sample will be collected for initial toxicity screening. Each sample will be collected at a time period selected to reflect discharge processes and operational processes. Collect and document initial toxicity screening samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP.

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- WET testing will be required if either of the following occurs: 1) Initial rapid toxicity screening threshold criteria are exceeded OR 2) discharge exceeds 10,000 gallons during any 24-hr period and chemicals are added to the system. If WET testing is required, collect and document samples in accordance with the procedures outlined in Section 2.4.3.1 of this QAPP. Immediately transfer the samples to the sample refrigerator for storage awaiting packaging for transportation to the fixed analytical laboratory. Package samples for transport to the fixed analytical laboratory in accordance with procedures outlined in this QAPP and M-I SWACO SOP 2003.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Collect and document a sample for static sheen test for discharge of bilge water (011) that has been processed through the OWS in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3004.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-22 summarizes the effluent monitoring tasks and frequencies for Discharge 011, Bilge Water.

Table 2-22 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 011

Frequency	Task	M-I SWACO (or Misc.) SOP
Daily	Document discharge flows and waste volumes	1006
	Visual sheen tests	1006, 3005
Four times per well	Initial effluent toxicity screening	1006, 2001, ENV001.01, TOX045.02
	WET testing (if any initial toxicity screening exceeds threshold criteria)	1006, 2001, Section 2.4.3, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Once per well	WET testing (if discharge exceeds 10,000 gallons during any 24-hour period and if chemicals are added to the system)	1006, 2001, TOX002.05, TOX012.06, TOX014B.02, TOX043.06
Monthly	pH sample	1006, 2001, 2012
Discharge event	Static sheen test	1006, 2001, 3004

Notes:
SOP = standard operating procedure
WET = whole effluent toxicity

2.4.3.13 Compliance Sampling for Excess Cement Slurry (Discharge 012)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

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- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Document the quantity of any chemical additive used.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks per discharge event (if conducted continuously):

- Perform and document visual sheen tests in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a monthly basis:

- Collect and document samples for pH analysis in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 2012. As soon as practicable, deliver pH samples to the rig compliance laboratory for analysis.

Table 2-23 summarizes the effluent monitoring tasks and frequencies for Discharge 012, Excess Cement Slurry.

Table 2-23 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 012

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
Monthly	pH sample	1006, 2001, 2012
Discharge event	Visual sheen test	1006, 2001, 3004

Notes:
SOP = standard operating procedure

2.4.3.14 Compliance Sampling for Muds, Cuttings, and Cement at the Seafloor (Discharge 013)

During active drilling operations, the M-I SWACO NPDES Compliance Specialist is responsible for performing the following tasks on a daily basis:

- Document the estimated flow volume in accordance with procedures outlined in this QAPP and M-I SWACO SOP 1006.
- Perform and document visual sheen tests for each outfall in accordance with procedures outlined in this QAPP and M-I SWACO SOPs 1006 and 3005.

Table 2-24 summarizes the effluent monitoring tasks and frequencies for Discharge 013, Muds, Cuttings, and Cement at the Seafloor.

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Table 2-24 Summary of Compliance Sampling, Measurement, and Observation Frequencies for Discharge 013

Frequency	Task	M-I SWACO SOP
Daily	Document discharge flows and waste volumes	1006
	Visual sheen tests	1006, 3005

Notes:
SOP = standard operating procedure

2.4.4 Field SOPs

The SOPs and LWIs are formal, revision-controlled documents that:

- Define the methods used in the performance of tasks having an effect on the quality of data, findings, or conclusions;
- Provide standard methods for execution and documentation of work, so as to maximize uniformity and reliability of products; and
- Facilitate coordination among individuals performing separate but interdependent tasks. SOPs and/or LWIs are generated through a cooperative effort among operations and QA personnel.

QA personnel coordinate their development, which involves an iterative process of review and revision until they are satisfactory to both QA and the technical reviewers.

2.4.4.1 M-I SWACO SOP and LWI Revisions

As a SOP or LWI is revised, the revision number is incremented and the revised SOP released to the distribution list. SOP and LWI revisions shall be processed through the M-I SWACO NPDES Compliance Supervisor or appropriate QA staff. SOP revisions may be necessitated by regulatory requirements, technological advancements, or other causes.

Dated acceptance signatures signify approval of the revisions. One technical reviewer and the M-I SWACO NPDES Compliance Supervisor or designee shall approve revisions of SOPs and LWIs. Once formally accepted, the revised document replaces the previous version and is distributed to SOP holders with instructions as to which document(s) it replaces.

The M-I SWACO NPDES Compliance Supervisor distributes SOPs and LWIs to technical staff and maintains distribution lists to confirm revisions. New SOPs and LWIs are distributed to responsible individuals.

2.4.4.2 Sampling SOPs and LWIs

Sampling SOPs and LWIs shall be developed, approved, and distributed to M-I SWACO NPDES Compliance Specialists. The SOP and/or LWI shall address, as applicable, the following elements:

- The objectives of the GP;
- A description of the overall sampling scheme (see below);

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- The number and location of sampling points;
- The number and location of control samples;
- Requirements for QC samples such as blanks, duplicates/replicates, matrix spikes, etc;
- The analyses to be performed (physical, chemical, toxicological, biological);
- The number and size (weight or volume) of samples to be collected for each type of analysis;
- Sample collection methods, specifying sampling equipment or SOPs, where applicable;
- Field screening procedures and criteria; and
- Sample containerization, preservation, and transportation procedures.

The M-I SWACO NPDES Compliance Specialists and subcontract laboratories shall be notified of sampling program schedules in advance, so that personnel and physical resources can be scheduled to meet sample holding time limitations and other GP requirements.

M-I SWACO operational discharge sampling-related SOPs and LWIs are listed in Table 2-25. The SOPs and LWIs listed in these tables are also included in Appendix A to this QAPP.

2.4.5 Analytical SOPs

Environ effluent toxicity testing SOPs are listed in Table 2-26 and included in Appendix A to this QAPP. Analytical SOPs for additional methods performed by subcontract laboratories are available upon request.

Table 2-25 Operational Discharge Compliance Sampling Related SOPs

M-I SWACO SOP Number	M-I SWACO SOP Title
1001	Qualification and Documentation of Computer Programs
1002	Traceability of Reagents, Standards, and Reference Materials
1003	Equipment Tracking
1004	Balance Calibration
1005	Laboratory Refrigerators
1006	Field Logs
1008	Demonstration of Capability
2001	Chain-of-Custody (C-O-C) Procedures
2002	Sample Collection for Suspended Particulate Phase (SPP) Toxicity Test
2003	Packaging and Shipment of Samples
2004	Decontamination of Equipment
2007	Sample Collection for Chromium VI, Silver, and Thallium in Water-Based Drilling Fluid
2008	Sample Collection for TAH and TAqH Analyses
2010	Sample Collection for Biochemical Oxygen Demand (BOD_5) Analysis

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M-I SWACO SOP Number	M-I SWACO SOP Title
2011	Sample Collection for Fecal Coliform Analysis
2012	Field Measurement of pH
2013	Field Measurement of Total Residual Chlorine
2014	Sample Collection for Total Suspended Solids (TSS) Analysis
3004	Free Oil by Static Sheen Method
3005	Visual Sheen Test Method
LWI-001	Continuous Temperature Monitoring
LWI-002	Chemical Inventory Management
LWI-009	Deployment of Overboard House
LWI-080	Pesticide/Biocide Handling/Mixing
LWI-080A	Mixing pesticide/biocide at the rig (Revision A)
LWI-080B	Mixing Pesticide/Biocides on the rig (Revision B)

Notes:

BOD_5 = biochemical oxygen demand
SOP = standard operating procedure
SPP = suspended particulate phase
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons
TSS = total suspended solids

Table 2-26 Drilling Fluid and Effluent Toxicity Testing SOPs

Environ SOP Number	Environ SOP Title
TOX002.05	7-Day Chronic Toxicity Study with *Atherinops affinis*
TOX012.064	7-Day Chronic Toxicity Study with *Menidia beryllina*
TOX014B.02	7-Day Chronic Toxicity Study with *Americamysis bahia* – Survival, Growth, and Fecundity
TOX043.06	Chronic Toxicity Test Using Echinoderm Larvae *(Strongylocentrotus purpuratus or Dendraster excentricus)*
TOX045.02	Chronic Toxicity Fertilization Test Using Echinoderms *(Strongylocentrotus purpuratus or Dendraster excentricus)*
ENV001.01	Chukchi Sea BES – Collection of Effluent Samples for Biological Testing

Notes:

SOP = standard operating procedure
SPP = suspended particulate phase

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2.4.6 Sample Handling and Custody

2.4.6.1 Responsibilities

The M-I SWACO NPDES Compliance Supervisor, or designee, is responsible for verifying that a sampling SOP is prepared that conforms to the provisions of this chapter, while fulfilling GP objectives. The M-I SWACO NPDES Compliance Supervisor is responsible for assembling or ordering rig compliance laboratory sampling kits. Fixed subcontract laboratories are responsible for supplying sample containers for fixed laboratory analyses and Barrow mobile laboratory analyses. M-I SWACO NPDES Compliance Specialists are responsible for collecting the field and QC samples as described in this chapter and as required by the GP; and for adhering to the sample packaging, labeling, and documentation requirements of this chapter and associated SOPs. The M-I SWACO NPDES Compliance Specialist is responsible for collecting grab samples at a point representative of the discharge. The subcontract laboratory sample custodian is responsible for the proper inspection, login, and storage of incoming samples, as defined in the laboratory's SOPs.

2.4.6.2 Sample Containers, Preservation, and Storage

In general, samples will be collected for rig compliance laboratory analysis, Barrow mobile laboratory analysis, and fixed subcontract laboratory analysis in containers as required by the analytical methods. Sample containers are provided by the laboratory and may be pre-cleaned and/or pre-preserved (if required by the analytical method).

The M-I SWACO NPDES Compliance Specialist will clean containers for static sheen and retort cuttings samples in the rig compliance laboratory following the procedures in SOP 2004 Decontamination of Equipment (Appendix A).

After collection, the samples are maintained at the required temperature (for most samples, <6°C) under CoC procedures until analysis or until they are shipped to a subcontract laboratory for analysis. For operational discharge compliance program samples, the samples should not be frozen. Procedures for packaging and shipping samples are outlined in M-I SWACO SOP 2003 Packaging and Shipment of Samples.

A summary of the analytical methods, sample containers, preservation, and holding times for samples that will be submitted to subcontract laboratories for analysis is included in Table 2-27.

Table 2-27 Sample Containers, Preservation, and Holding Times

Analyte/ Analyte Group	Method/SOP	Container(s) (number, size, & type per sample)	Preservation	Holding Time
OIL MATRIX				
Diesel fingerprint	SW8015C	(2) 40-mL VOA vials w/TLS	Cool <6°C	7 days to extraction, 40 days to analysis
AQUEOUS MATRIX				
TAH/TAqH[1]	E624	(3) 40-mL VOA vials w/TLS	Cool <6°C, HCl to pH<2	14 days
	E625 SIM	(2) 1-L amber glass jars w/TLC	Cool <6°C	7 days to extraction, 40 days to analysis

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Analyte/ Analyte Group	Method/SOP	Container(s) (number, size, & type per sample)	Preservation	Holding Time
Diesel fingerprint	SW8015C	(2) 40-mL VOA vials w/TLS	Cool <6°C	7 days to extraction, 40 days to analysis
SPP toxicity	40 CFR 435, App. 2 to Subpart A (E1619)	(1) 1-gal (4L) LDPE jar	0 to 4°C	90 days
Initial toxicity screening	EPA/600/R-95-136 (Echinoderm Fertilization Test)	(1) 1-L LDPE bottle	Cool <6°C	36 hours (72 hours maximum)
WET[2]	EPA/600/R-95-136 (Topsmelt Chronic 7d Survival and Growth Test)[1] or EPA/821-R-02-014 (Menidia Chronic 7d Survival and Growth Test)[1]	(3) 10-L LDPE cubitainers	Cool <6°C	36 hours (72 hours maximum)
	EPA/821-R-02-014 (Mysid Chronic 7d Survival, Growth, and Fecundity Test)[1]	(3) 10-L LDPE cubitainers	Cool <6°C	36 hours (72 hours maximum)
	EPA/600/R-95-136 (Echinoderm Larval Development Test)	(1) 1-L LDPE bottle	Cool <6°C	36 hours (72 hours maximum)
BOD_5	SM5210-B	(1) 1-L poly bottle	Cool <6°C	48 hours
TSS	SM2540-D	(1) 1-L poly bottle	Cool <6°C	7 days
Fecal coliform bacteria	SM9222-D	(1) 125-mL sterile poly container w/ $Na_2S_2O_3$	Cool <6°C, Na_2SO_3	6 hours

Notes:

[1] Samples for these tests may be provided as one, 10-L sample on an every-other-day basis. If this sample strategy is not feasible, a single sample of 30 L may be provided at one time.

[2] Samples for WET testing may be collected in larger containers (up to 20-L LDPE cubitainers or different combination thereof) to facilitate sufficient volume to conduct all three tests.

BOD5 = biochemical oxygen demand
HCl = hydrochloric acid
mL = milliliter
L = liter
Na2S2O3 = sodium thiosulfate
SOP = standard operating procedure
SPP = suspended particulate phase
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons
TLC = Teflon-lined cap
TLS = Teflon-lined septa
TSS = total suspended solids
VOA = volatile organic analysis
WET = whole effluent toxicity

2.4.6.3 Sample Receipt

Sample receipt and CoC will be maintained at the fixed subcontract laboratories in accordance with the procedures outlined in Section 1.8 of this QAPP. Drilling and effluent samples for toxicity testing require additional procedures to verify acceptability upon receipt at the fixed laboratory.

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Receipt of Drilling Fluid Samples for SPP Toxicity Analysis

Drilling fluid samples provided for biological testing must also meet the temperature requirements described above. The pH of the drilling fluid samples will be tested upon receipt at the laboratory. If the pH is less than 9, if black spots have appeared on the walls of the sample container, or if the mud sample has a foul odor, then the sample does not meet the method specific acceptability criteria for drilling fluids as specified in 40 CFR Part 435. If the drilling fluid does not meet the acceptability criteria for testing, the client will be notified and the decision to proceed will be evaluated. The drilling fluid should be used for testing within three months from the time of collection. Elutriate samples prepared at the laboratory from the drilling fluids should be used for testing within the same day.

Receipt of Effluent Samples for Initial Toxicity Screening and WET Testing Samples

Upon receipt at the subcontract laboratory, water quality parameters are measured for effluent samples. These parameters include dissolved oxygen, temperature, pH, and salinity/conductivity. Acceptability criteria for water quality parameters are included in Section 2.6.

2.4.6.4 Holding Times

Sample holding time begins with the collection of the sample and continues until the analysis is complete. Analytical method holding times are included in Table 2-27.

Testing for toxicity samples should be initiated on samples within 36 hours of sample collection, but must not exceed 72 hours as prescribed in the GP and method guidance. Effluent samples used for test solution renewals on the WET 7-day chronic tests (days 1-6) may be used up to 48-hours after the initial use (test initiation) and may be used for test solution renewals at up to 120 hours from the time of collection.

2.4.6.5 Sample Retention and Disposal

Procedures ensuring internal laboratory chain-of-custody shall also be implemented and documented by the subcontract laboratory. Samples shall be stored in limited-access, temperature-controlled areas. Samples will be retained and disposed of in accordance with laboratory procedures.

Samples will be retained under proper storage conditions in the rig compliance laboratory. Static sheen samples will be retained until the end of the holding time or until the M-I SWACO NPDES Compliance Specialist verifies the acceptability of the sample results. In the event a sample result shows a permit exceedance, the sample will be retained until the permittee requests additional testing or provides permission for disposal. After expiration of the sample retention period, mud samples are returned to the active mud system and cuttings samples are placed in the cuttings disposal system.

2.4.6.6 Decontamination

Decontamination is essential for the avoidance of cross-contamination among samples. Reusable, non-dedicated sampling implements are decontaminated before use and after samples are collected, in accordance with M-I SWACO SOP 2004 Decontamination of Equipment. Generally, sample equipment used to collect samples for GP compliance is dedicated equipment and requires minimal decontamination.

2.4.7 Field Instrument/Equipment Testing, Inspection, Calibration, and Maintenance

Effluent limitations in the GP involve measurements that shall be made directly in the field –for example, the Free Oil by Static Sheen Method (M-I SWACO SOP 3004). In addition, effluent limitations in the GP involve measurements that are made in an analytical laboratory – for example, TAH/TAqH and toxicity testing. These measurements involve the use of instrumentation that shall be calibrated and in good working condition in order to fulfill the quality objectives of the GP.

This section discusses M-I SWACO's program for calibration and verification of measuring and test equipment used to provide compliance data for the GP. Specific procedures for operation, maintenance, and calibration of rig compliance laboratory instrumentation can be found in the associated M-I SWACO SOP and/or manufacturer's instructions for each instrument.

The fixed subcontract laboratory QA Manual documents the quality system under which the laboratory operates. The testing, inspection, calibration, and maintenance activities for analytical laboratory instrumentation are documented in the subcontract laboratory's QA Manual and SOPs.

2.4.7.1 Responsibilities

The M-I SWACO NPDES Compliance Specialist or designee will be responsible for maintaining, issuing, and tracking field equipment in accordance with this chapter and relevant M-I SWACO SOPs. The Shell Environmental Compliance Engineer is responsible for designing QC programs for field measurements. M-I SWACO NPDES Compliance Specialists are responsible for performing field measurements, and maintaining and calibrating field equipment in accordance with M-I SWACO SOPs, this QAPP, and the GP, and for accurately completing the attendant documentation.

The storage, preventive maintenance, issuing, and tracking of equipment are the responsibility of the M-I SWACO NPDES Compliance Supervisor. The M-I SWACO NPDES Compliance Supervisor or designee performs the following tasks:

- Storage of Equipment - Field equipment is kept in a designated, limited-access storage area.
- Controlling access to the storage area.
- Issuing Equipment - Field equipment is issued by the M-I SWACO NPDES Compliance Supervisor.
- Preventive Maintenance and Repair – The M-I SWACO NPDES Compliance Specialist performs preventive maintenance of field equipment in accordance with the manufacturers recommended schedule and procedures.
- Record keeping and tracking – The M-I SWACO NPDES Compliance Supervisor maintains one Equipment Manual for each model of equipment in the Area Office. The M-I SWACO NPDES Compliance Supervisor also keeps records of routine preventive maintenance, repairs and utilization of field equipment in an Equipment Log. Each time a piece of equipment is repaired, or routine maintenance is performed, an entry is made in the logbook, giving the date, nature of repair or maintenance, identification (description and serial number) of the equipment and the initials of the person performing the repairs or maintenance work.

Subcontract laboratories are responsible for performing analytical measurements and maintaining and calibrating equipment in accordance with laboratory SOPs, the laboratory QA Manual, applicable analytical methods, and the GP; and for accurately completing the attendant documentation. Subcontract laboratory documentation shall be available upon request for audit or inspection, and equipment and reference material records shall be maintained in accordance with laboratory record retention policy.

2.4.7.2 Rig Compliance Laboratory Equipment

Sampling and analytical procedures are performed at the permittee's drilling rig using M-I SWACO rig compliance laboratories. The M-I SWACO NPDES Compliance Specialist shall provide sampling equipment to safely collect representative samples of drilling fluids and drill cuttings and other effluents. The M-I SWACO NPDES Compliance Specialist shall also provide analytical equipment and supplies necessary to perform the Free Oil by Static Sheen Method (M-I SWACO SOP 3004).

M-I SWACO shall maintain duplicate equipment for the Free Oil by Static Sheen Method (M-I SWACO SOP 3004) test at the rig compliance laboratory.

The permittee shall provide M-I SWACO with access to a sample refrigerator that is capable of maintaining the drilling fluid and drill cutting samples at < 6 degrees Celsius (°C). The permittee shall provide a well-ventilated work area with sufficient counter space and adequate fluorescent lighting to perform procedures. Electricity shall be of sufficient voltage and amperage to operate retorts, balances, computers, and ultraviolet light. The permittee shall also provide storage space for the flammable solvent, isopropanol.

2.4.7.3 Field and Rig Compliance Laboratory Instrument Calibration

Instrument calibration comprises initial calibration that is used directly for quantification and continuing calibration verification that is used to confirm the validity of the initial calibration. Analytical procedures for the rig compliance laboratory, such as pH analysis, may require initial calibration and/or calibration verification. Analytical procedures for the fixed laboratory analyses may require initial and continuing calibration. Calibration procedures will be described in the applicable laboratory SOPs.

Calibration of thermometers and balance weights shall be verified every two years using National Institute of Standards and Technology (NIST) standards. Each working day, balances and refrigerators, shall be checked in the expected use range with NIST traceable standards. Checks shall be recorded on the equipment logs provided in the applicable SOPs. The equipment logs shall indicate the acceptable range for the check. Corrective actions for calibration results outside the acceptance range shall also be recorded on the equipment logs. If a corrective action is not successful, then the equipment shall be removed from service.

<u>Calibration Procedures</u>

Equipment shall be calibrated at the frequency specified in the applicable SOPs. Equipment shall be calibrated using the procedures specified in the applicable SOPs. The appropriate form for recording the calibration of each device is provided in the applicable SOP. The status of equipment with regard to calibration needs to be readily available and clearly indicated on equipment and field logs. Applicable acceptance criteria for equipment inspection and calibration shall be met before any sample collection or

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sample analyses are performed. Specific procedures for equipment calibration shall be documented in the relevant SOP. The results of the inspection and calibration shall be documented on Equipment Inspection and/or Calibration Forms.

Calibration Verification

Equipment calibration shall be verified by the analysis of appropriate reference materials before samples are collected /analyzed and at the required frequency throughout collection and/or analysis. The results shall be within the acceptance limits for the applicable SOP in order to proceed with sample collection and/or analysis.

Calibrations shall be documented on the Equipment Log (in the M-I SWACO Area Office) or Equipment Calibration Form (on the Drilling Rig). Records pertaining to calibration shall be safely stored for the period described in M-I SWACO's record retention policy. The following information shall be recorded:

- Date;
- Time;
- Instrument type and serial number;
- Identification of calibration standard;
- True value (numerical result, positive, negative) of calibration standard;
- Response of instrument before repair or adjustment;
- Response of instrument after repair or adjustment; and
- Signature of person performing the calibration.

Rig Compliance Laboratory Equipment Calibration

Portable analytical instruments (e.g. retorts, balances, and ultraviolet lights) require preventive and corrective maintenance, as well as calibration. Calibrations shall be performed in accordance with the appropriate M-I SWACO SOP and/or manufacturer's instructions. Portable equipment shall be calibrated and inspected prior to and upon return from the field. Rig compliance laboratory equipment calibration is summarized in Table 2-28.

Table 2-28 Onsite Laboratory Equipment Calibration

Equipment	Calibration Procedure	Calibration Verification		SOP
		Method	Frequency	
Balance	Manufacturer's specifications	Calibration check – 100 g, 500 g, 1000 g, and 2000 g F Class Calibration Weights	Daily	1004
pH Meter	Manufacturer's Specification	Calibration check by buffer solution(s)	Daily	2012
Refrigerator	Manufacturer's specifications	Verification of operating temperature by certified thermometer	Daily	1005

Notes:
g = grams SOP = standard operating procedure

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Complete calibration verification forms as indicated in applicable SOPs. Forms will be maintained on drilling rig until completion of drilling season. After completion of drilling season, forms will be archived in the M-I SWACO Area Office.

2.4.7.4 Standards and Reagents

The M-I SWACO NPDES Compliance Specialists shall retain records for standards and reagents, including the manufacturer/vendor, the Certificate of Analysis or purity, the date of receipt, recommended storage conditions, and an expiration date. Original containers shall be labeled with an expiration date and a unique standard or reagent identifier. The standard or reagent identifier shall also be recorded on the Certificate of Analysis or vendor documentation.

Standards and reagents records shall include the purchased stock or neat compound identifier number, a reference to the method of preparation (such as a SOP number) or a description of the preparation (including weights and/or volumes), the date of preparation, the expiration date and the preparer's initials. Each prepared standard and reagent will be assigned a unique identifier that is linked to the documentation described in this paragraph. The container for each standard and reagent will be labeled with the unique identifier and the material expiration date. In order to protect the health and safety of personnel, containers will be labeled with the Hazardous Materials Identification System (HMIS) information.

2.4.7.5 Reference Materials and Standards

Equipment and standards shall be verified before use and on a continuing basis. Traceability and calibration of fixed laboratory equipment will be discussed in the subcontracted laboratory QA Manual. Documentation will be maintained at the fixed laboratory as discussed in Section 1.

Reference Materials

Reference materials are materials or substances for which one or more properties are sufficiently well established to be used for the calibration of an apparatus, for assessment of methods or for assigning values to materials. Generally reference materials are purchased from third parties such as NIST and are characterized for content, independent of an analytical method. Reference materials used to support data generated for permit compliance include:

- Class F weights
- NIST thermometers

Original certificates are maintained at the M-I SWACO Area Office and certificate copies are maintained with equipment in the rig compliance laboratory. Reference materials are removed from service upon expiration of the certificate and may only be placed back into service once the material is recertified.

Reference Standards

Reference standards shall be used for calibration only. Reference standards are removed from service upon expiration of the certificate or if calibration indicates the standard is outside of specifications. If the material is outside of specifications and correction factors can be established, the reference standard may

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continue in service. The M-I SWACO Area Office shall maintain original records of the correction factors with the applicable dates. Copies of relevant correction factors shall accompany the material in the field.

Traceability

Calibration certificates shall be provided by the manufacturer and shall indicate traceability to national standards. Class F weights, crude oil standards, and NIST thermometers are examples of materials for which calibration certificates shall be maintained. Certificates that describe purity or composition of standard materials shall also be maintained. Certificates shall be cross-referenced to a material identification, such as equipment number, standard, or reagent number.

2.4.7.6 Refrigerator Testing

Refrigerators will be monitored using thermometers that have been calibrated using a NIST-traceable thermometer. Refrigerator temperatures will be monitored daily and the temperature recorded on Form 1005-2 Refrigerator Calibration Log. Thermometer calibration procedures and refrigerator monitoring procedures are included in SOP 1005 Laboratory Refrigerators.

2.4.7.7 Field Compliance Instrument/Equipment Maintenance

Field equipment is maintained under a tracking and preventive maintenance program. When a piece of permit-related equipment is sent to a vendor for repairs or maintenance, a log entry is made giving the date, name of vendor, identification of the equipment, description of services required, and initials of the person sending the equipment. When the equipment is returned, a similar entry is made to document the return and a copy of the repair slip is placed in the Equipment Log.

When equipment is issued, an entry is made in the Equipment Log giving the date, identification of the equipment, initials of the person issuing the equipment and the identity of the person to whom the equipment is issued. When the equipment is returned, a similar entry is made, noting date of return, and condition of the returned equipment. Through the use of the log, the M-I SWACO NPDES Compliance Supervisor maintains a continuous written record of the status and the location of each piece of field equipment.

Equipment that is overdue for scheduled maintenance or is in need of repair is placed “Out of Service”. This equipment shall be clearly identified as “Out of Service” until the repair work has been completed and the equipment is operating satisfactorily. Equipment that is not marked shall be considered in proper working order.

2.4.8 Fixed Subcontract Laboratory Instrument/Equipment Testing, Inspection, Calibration, and Maintenance

Testing, inspection, calibration, and maintenance for fixed laboratory equipment is performed in accordance with laboratory QA Manuals and analytical method requirements. Calibration requirements for fixed laboratory equipment are documented in subcontract laboratory SOPs. Subcontract laboratories shall maintain equipment and have in place either a service contract, or sufficient spare parts, such that method holding times or analytical report due dates are not exceeded.

The following sections outline specific equipment, materials, and procedures used by the toxicity testing subcontract laboratory.

2.4.8.1 Initial Toxicity Screening and Whole Effluent Toxicity Testing Laboratory Equipment Washing Procedures

All laboratory equipment, glassware, and plasticware used for the toxicity testing program will be cleaned to eliminate potential toxicity associated with labware. Plastic and glassware are scrubbed with a solution of non-phosphate detergent and hot tap water, and then rinsed three times with deionized water. A rinse of 10% solution reagent grade hydrochloric acid is followed by three rinses with deionized water. A rinse of reagent grade acetone is followed by air drying under a fume hood. Acetone is not used on hard plastics. A final three rinses with deionized water is followed by air drying.

2.4.8.2 Initial Toxicity Screening and Whole Effluent Toxicity Testing Laboratory Materials

Test Organisms Suppliers

Test organisms used in the toxicity tests will be from either in-house cultures or collected in areas known to be generally free of pollutants, or purchased from reputable culturists. Organisms are purchased from suppliers who are selected based on their reputation, depth of knowledge concerning the test organisms, and their ability to consistently deliver healthy test organisms.

Upon receipt in the laboratory, test organisms will be slowly acclimated to test conditions in environmentally controlled holding areas in accordance with the test protocol for each test organism. Test organisms will be evaluated on a performance basis for every test conducted in the laboratory. Negative controls will be tested concurrent to each study to evaluate the health of the test organisms and the acceptability of the test conditions. Positive controls (reference toxicant tests) will also be conducted to assess the relative sensitivity of test organisms compared to historical laboratory performance.

Source of Seawater

Seawater diluent that will be used in this study will come from the northern Hood Canal at Port Gamble, Washington. This water source (used as the laboratory control and sample diluent) has been used successfully on a wide range of bioassay testing programs. Artificial seawater may also be prepared from deionized water and bioassay-grade sea salts (Crystal Sea Marinemix™) if necessary. Extensive testing with a variety of test species has shown that there is no significant potential for toxicity or bioaccumulation from these water supplies. Chemical analyses are performed annually on these water sources and have shown no significant contaminants of concern or bioaccumulation potential.

2.5 Fixed Laboratory Analysis

The fixed subcontract laboratories that are proposed for use for the analysis of compliance monitoring samples are listed for the corresponding preparation and analytical methods in Table 2-29.

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Table 2-29 Analytical Subcontract Laboratories

Analytical Parameter	Matrix	Preparation/ Analytical Method	Laboratory/Organization
TAH/TAqH	Aqueous	SW5030B/E624 SW3520C/E625 SIM	Primary Laboratory: SGS North America, Inc. 200 W. Potter Drive Anchorage, AK 99518 (907) 562-2343 Main (907) 561-5301 Fax Backup laboratory: TestAmerica Anchorage 2000 W. International Airport Rd. Suite A10 Anchorage, AK 99502 (907) 563-9200 Main (907) 563-9210 Fax
Diesel fingerprinting	Aqueous Oil	SW3520C/SW8015C SW3550C/SW8015C	
BOD_5	Aqueous	SM5210-B	
TSS	Aqueous	SM2540-D	
Fecal coliform	Aqueous	SM9222-D	Primary Laboratory: SGS Mobile Laboratory Barrow, AK 99723 Backup Laboratories: SGS North America, Inc. 200 W. Potter Drive Anchorage, AK 99518 (907) 562-2343 Main (907) 561-5301 Fax TestAmerica Anchorage 2000 W. International Airport Rd. Suite A10 Anchorage, AK 99502 (907) 563-9200 Main (907) 563-9210 Fax Arctic Fox Environmental, Inc. Pouch 340043 Prudhoe Bay, AK 99734 (907) 659-2145 Main (907) 659-2146 Fax
SPP toxicity	Aqueous	40 CFR Part 435, App. 2 to Subpart A (E1619)	Primary Laboratory: Environ 4729 NE View Drive Port Gamble, WA 98364 (360) 297-6040 Main Backup Laboratories: Environmental Enterprises USA, Inc. 58485 Pearl Acres Road, Suite D Slidell, LA 70461 (800) 966-2788 Main (985) 646-2810 Fax Element Materials Technology 2417 West Pinhook Road Lafayette, LA 70508 (337) 235-0483 Main (337) 233-6540 Fax
Initial toxicity screening	Aqueous	EPA/600/R-95-136 (Echinoderm Fertilization Test)	
WET testing	Aqueous	EPA/600/R-95-136 (Topsmelt Chronic 7d Survival and Growth Test) or EPA/821-R-02-014 (Menidia Chronic 7d Survival and Growth Test)	
		EPA/821-R-02-014 (Mysid Chronic 7d Survival, Growth, and Fecundity Test)	
		EPA/600/R-95-136 (Echinoderm Larval Development Test)	

Notes:

BOD_5 = biochemical oxygen demand
SPP = suspended particulate phase
TAH = total aromatic hydrocarbons
TAqH = total aqueous hydrocarbons

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TSS = total suspended solids WET = whole effluent toxicity

2.5.1 Analytical Laboratory Methods

Evaluation of the effluent limits and laboratory quantitation limits is required to ensure that project data quality objectives are met. Laboratory-specific detection limits and reporting limits will be evaluated against the permit effluent limitations to determine whether the sensitivity of the data will be sufficient for its intended use.

Proposed drilling fluid and effluent toxicity testing methods are summarized in Table 2-30. The initial toxicity screening of effluent discharges will utilize the Chronic Toxicity Fertilization Test using echinoderms (*Strongylocentrotus purpuratus* or *Dendraster excentricus*) as outlined in the "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to West Coast Marine and Estuarine Organisms" (EPA/600/R-95-136).

The methods for WET testing are provided in established EPA procedures outlined in "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to Marine and Estuarine Organisms" (EPA-600-4-91-003-Third Ed.) and the "Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to *West Coast* Marine and Estuarine Organisms" (EPA/600/R-95-136). The WET testing program will use three different species of organisms including the topsmelt, *Atherinops affinis (or M. beryllina*-depending on availability*)*, the mysid shrimp, *Americamysis bahia*, and the purple sea urchin, *Strongylocentrotus purpuratus*.

Table 2-31 presents subcontract laboratory analytical methods, detection limits, and reporting limits for effluent monitoring.

Table 2-30 Drilling Fluid and Effluent Toxicity Testing Methods

Toxicity Test	Test Description	Species	Method
Drilling fluid SPP toxicity	Mysid 96-Hour Survival Test	*Americamysis bahia* (Formerly *Mysidopsis bahia*)	40 CFR Part 435 EPA-821-R-11-004 EPA-821-R-02-012
Initial (Rapid) toxicity screening test	Chronic Toxicity Echinoderm Fertilization Test	Purple Sea Urchin *(Strongylocentrotus purpuratus)* or Sand Dollar *(Dendraster excentricus)*	EPA/600/R-95/136
Marine Chronic Toxicity Test (WET testing)	Larval Fish 7-Day Larval Survival and Growth Test	Topsmelt (Atherinops affinis) or Inland Silverside[1] (Menidia beryllina)	EPA/600/R-95/136 EPA-821-R-02-014
	Mysid Shrimp 7-Day Larval Survival, Growth, and Fecundity Test	*Americamysis bahia* (Formerly *Mysidopsis bahia*)	EPA-821-R-02-014
	Echinoderm Larval Survival and Development Test	Purple Sea Urchin (*Strongylocentrotus purpuratus*) or Sand Dollar (*Dendraster excentricus*)	EPA/600/R-95/136

Notes:

[1] *Menidia beryllina* may be used as a substitute for topsmelt.
EPA = U.S. Environmental Protection Agency WET = whole effluent toxicity
SPP = suspended particulate phase

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Table 2-31 Analytical Laboratory Reporting Limits

Analytical Method	Analyte	Matrix	Permit Limit	Units	DL	RL
SM5210-B	BOD_5	Aqueous	30	mg/L	2	2
SM2540-D	TSS	Aqueous	30	mg/L	0.15	0.5
SM9222-D	Fecal coliform	Aqueous	100	col/100 mL	1	1
E624	Benzene	Aqueous	NA	µg/L	0.12	0.4
E624	Toluene	Aqueous	NA	µg/L	0.31	1
E624	Chlorobenzene	Aqueous	NA	µg/L	0.15	0.5
E624	Ethylbenzene	Aqueous	NA	µg/L	0.31	1
E624	P & M -Xylene	Aqueous	NA	µg/L	0.62	2
E624	o-Xylene	Aqueous	NA	µg/L	0.31	1
E624	1,3-Dichlorobenzene	Aqueous	NA	µg/L	0.31	1
E624	1,4-Dichlorobenzene	Aqueous	NA	µg/L	0.15	0.5
E624	1,2-Dichlorobenzene	Aqueous	NA	µg/L	0.31	1
E625 SIM	Acenaphthene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Acenaphthylene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Anthracene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Benzo[a]anthracene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Benzo[a]pyrene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Benzo[b]Fluoranthene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Benzo[g,h,i]perylene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Benzo[k]fluoranthene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Chrysene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Dibenzo[a,h]anthracene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Indeno[1,2,3-c,d] pyrene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Fluoranthene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Fluorene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Naphthalene	Aqueous	NA	µg/L	0.031	0.1
E625 SIM	Phenanthrene	Aqueous	NA	µg/L	0.015	0.05
E625 SIM	Pyrene	Aqueous	NA	µg/L	0.015	0.05
SW8015C	Diesel Range Organics	Aqueous	NA	mg/L	0.15	0.4
SW8015C	Diesel Range Organics	Oil	NA	mg/kg	620	2000

Notes:

SGS North America, Inc. DLs and RLs.
BOD_5 = biochemical oxygen demand
DL = detection limit
mg/kg = milligrams per kilogram
mg/L = milligrams per liter
µg/L = micrograms per liter
RL = reporting limit
SPP = suspended particulate phase
TSS = total suspended solids
TU_c = chronic toxic units

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2.6 Field and Laboratory Quality Control Samples

2.6.1 Field Quality Control Samples and Frequencies

Field QC samples that will be collected and/or submitted include field duplicates, MS/MSDs, trip blanks, and temperature blanks. Field QC samples will be collected and/or submitted at the following frequencies:

- Field duplicates will be collected at a frequency of one per 20 or fewer primary samples for each analyte and matrix as described in Table 2-32.
- MS and MSD samples will be collected for every 20 or fewer samples as described in Table 2-32.
- Trip blanks will be submitted with every cooler for every shipment containing volatile samples.
- Temperature blanks will be submitted with every cooler containing analytical samples.

Table 2-32 summarizes the type and quantity of QC samples required for compliance monitoring.

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Table 2-32 Field Quality Control Samples

Discharge	Matrix	Monitoring Parameter	Method	Sampling Frequency	Field Duplicate	MS/MSD[1]	Trip Blank Required?
Discharge 001 Water-based drilling fluids and drill cuttings	Water-based drilling fluids and drill cuttings	SPP toxicity	E1619	Weekly and end of well	Duplicate collected each time a primary sample is collected[2]	NA	NA
		TAH	E624	Once per well	One duplicate per 20 or fewer samples	One MS/MSD per 20 samples	Yes
		TAqH	E625 SIM	Once per well	One duplicate per 20 or fewer samples	One MS/MSD per 20 samples	NA
		Diesel oil (Diesel-range organics)	SW8015C	Once per well, or if static sheen test fails	One duplicate per 20 or fewer samples	One MS/MSD per 20 samples	NA
	Diesel oil in storage	Diesel oil (Diesel-range organics)	SW8015C	If static sheen test fails	NA	NA	NA
Discharge 002 Deck Drainage	OWS effluent	TAH	E624	Once per discharge event (if conducted continuously)	One duplicate per 20 or fewer samples	One MS/MSD per 20 samples	Yes
	OWS effluent	TAqH	E625 SIM	Once per discharge event (if conducted continuously)	One duplicate per 20 or fewer samples	One MS/MSD per 20 samples	NA
Discharge 003 Sanitary Wastes	MSD effluent	BOD_5	SM5210-B	Weekly	NA	NA	NA
	MSD effluent	TSS	SM2540-D	Weekly	NA	NA	NA
	MSD effluent	Fecal coliform bacteria	SM9222-D	Weekly	NA	NA	NA

Notes:

[1] Triple volume of sample must be collected for MS/MSD for organic analyses. Two sample volumes must be collected for inorganic analyses.

[2] A duplicate sample volume will be collected with each primary sample. The duplicate volume will be retained in the rig compliance laboratory refrigerator until acceptable results are received for the sample transported to the laboratory. If a problem occurs with the original sample that invalidates the results, the duplicate volume of sample will be packaged and transported to the subcontract laboratory for analysis.

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2.6.2 Laboratory Quality Control

2.6.2.1 Analytical Method Quality Control Criteria

Quality control checks for sample collection will be accomplished by a combination of CoC protocols, field QA samples, and laboratory QC as prescribed in the sampling or analytical methods. Fixed laboratory QC samples will be prepared and analyzed as required by the analytical methods and laboratory QA Manuals. Results from LCS/LCSD and MS/MSD samples will be compared to criteria for accuracy and precision listed in Table 2-33.

Table 2-33 Analytical Method Quality Control Criteria

Analytical Method	Analyte	Matrix	Laboratory Control Limits (Accuracy)		RPD (Precision)
			LCL (%)	UCL (%)	
SM5210-B	BOD_5	Aqueous	84.6	115.4	20
SM9222-D	Fecal coliform	Aqueous	NA	NA	NA
SM2540-D	TSS	Aqueous	75	125	5
E624	Benzene	Aqueous	80	120	20
E624	Toluene	Aqueous	75	120	20
E624	Chlorobenzene	Aqueous	80	120	20
E624	Ethylbenzene	Aqueous	75	125	20
E624	P & M -Xylene	Aqueous	75	130	20
E624	o-Xylene	Aqueous	80	120	20
E624	1,3-Dichlorobenzene	Aqueous	75	125	20
E624	1,4-Dichlorobenzene	Aqueous	75	125	20
E624	1,2-Dichlorobenzene	Aqueous	70	120	20
E625 SIM	Acenaphthene	Aqueous	45	110	30
E625 SIM	Acenaphthylene	Aqueous	50	105	30
E625 SIM	Anthracene	Aqueous	55	110	30
E625 SIM	Benzo[a]anthracene	Aqueous	55	110	30
E625 SIM	Benzo[a]pyrene	Aqueous	55	110	30
E625 SIM	Benzo[b]Fluoranthene	Aqueous	45	120	30
E625 SIM	Benzo[g,h,i]perylene	Aqueous	40	125	30
E625 SIM	Benzo[k]fluoranthene	Aqueous	45	125	30
E625 SIM	Chrysene	Aqueous	55	110	30
E625 SIM	Dibenzo[a,h]anthracene	Aqueous	40	125	30
E625 SIM	Indeno[1,2,3-c,d] pyrene	Aqueous	45	125	30
E625 SIM	Fluoranthene	Aqueous	55	115	30
E625 SIM	Fluorene	Aqueous	50	110	30
E625 SIM	Naphthalene	Aqueous	40	100	30
E625 SIM	Phenanthrene	Aqueous	50	115	30
E625 SIM	Pyrene	Aqueous	50	130	30
SW8015C	Diesel Range Organics	Aqueous	75	125	20

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Analytical Method	Analyte	Matrix	Laboratory Control Limits (Accuracy)		RPD (Precision)
			LCL (%)	UCL (%)	
SW8015C	Diesel Range Organics	Oil	NA	NA	NA

Notes:
BOD5 = biochemical oxygen demand
LCL = lower control limit
RPD = relative percent difference
TSS = total suspended solids
UCL = upper control limit
WET = whole effluent toxicity

For operational discharge compliance sampling under the GP, the completeness goal is 100%. If a sample is determined to be invalid or unusable, the sample will be recollected as soon as practicable. The M-I SWACO NPDES Compliance Specialist will be responsible for ensuring that all samples are collected as required by the permit.

2.6.2.2 Toxicity Testing Quality Control Criteria

The test acceptability criteria and performance standards for the proposed toxicity tests are summarized in Tables 2-34 and 2-35. The toxicity tests incorporate standard QA/QC procedures to ensure that the test results are valid. Standard QA/QC procedures include the use of negative and positive controls, the use of testing replicates, and water quality monitoring. All limits established for this program meet or exceed those recommended by USEPA.

All data collected and produced will be recorded on approved data sheets, which will become part of the permanent data record of the program. If any aspect of a test deviates from protocol, the test will be evaluated to determine whether it is valid according to test acceptability criteria and performance standards.

Each toxicity test includes sample replication of 3 to 8 replicates, depending on the test. The data generated are checked by the responsible laboratory technician and reviewed independently by another analyst to assess precision. The maximum percent difference (MPD) for replicates and the reference toxicant tests are evaluated relative to the permissible bounds of the methods. Acceptable accuracy levels are also assessed by the calibration of water quality instruments, the use of certified standards, and the establishment of acceptable water quality testing parameters. For example, water quality is monitored and, adjusted if necessary, throughout testing in at least one test replicate. Parameters that fall outside of acceptable test ranges may require corrective action.

The sensitivity of test organisms will be evaluated using positive control reference toxicant tests. Reference toxicant tests will be conducted on each batch of test organisms either concurrently with project samples or monthly, as specified under the QA requirements for each test. The reference toxicant test will consist of an exposure to at least five concentrations of a reference substance (known toxicant) used to assess the health and sensitivity of the test organisms. The test duration and endpoint will mimic those of the corresponding bioassay. Dilutions will be selected to bracket laboratory historical LC_{50}/EC_{50}. The LC_{50}/EC_{50} results will be compared with historical data from definitive bioassays with

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the reference substance. The reference toxicant substances will be copper sulfate, ammonium chloride, or other toxicants as appropriate for evaluating the sensitivity of test organisms.

Table 2-34 Water Quality Criteria and Performance Standards

Analyte	Water Quality Test Parameters Acceptability Criteria			
	Dissolved oxygen	**Temperature**	**pH**	**Salinity**
Drilling Fluid (Mud) SSP Mysid Acute Survival Toxicity Test	Aeration to maintain DO > 5.3 mg/L	20 ± 2°C	6 – 9[3]	20 ± 2 ppt
Chronic Toxicity Echinoderm Fertilization Test	DO > 4.0 mg/L	12 ± 1°C	6 – 9	30 ppt[1]
Larval Fish 7-Day Chronic Survival and Growth Test	Aerate if DO < 4.0 mg/L	20 ± 1°C	6 – 9	5 – 34 ppt[1]
Larval Fish 7-Day Chronic Survival and Growth Test	Aerate if DO < 4.0 mg/L	25 ± 1°C	6 – 9	5 – 32 ppt[1]
Mysid Shrimp 7-Day Larval Survival, Growth, and Fecundity Test	Aerate if DO < 4.0 mg/L	26 ± 1°C	6 – 9	20 – 30 ppt[1]
Echinoderm Larval Survival and Development Test	Aerate if DO < 4.0 mg/L[2]	15 ± 1°C	6– 9	32 ± 2 ppt

Notes:

[1] Target should not deviate by ± 2 ppt during test period.

[2] No aeration during testing.

[3] 7.8 ± 0.1 (at initiation in the 100% elutriate).

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Table 2-35 Toxicity Test Acceptance Criteria and Performance Standards

Test Organism (Protocol – SOP)	Test Acceptability Criteria
Drilling Fluid (Mud) SPP Mysid Acute Survival Toxicity Test (40 CFR Part 435, EPA-821-R-11-004, EPA-821-R-02-012 – SOP No. SED026.01)	**1.** Survival in the controls ≥ 90%
Initial Toxicity Screening Test	
Chronic Toxicity Echinoderm Fertilization Test (*Strongylocentrotus purpuratus* or *Dendraster excentricus*) (EPA/600/R-95/136, WDOE WQ-R-95-80 – SOP No. TOX045.02)	**1.** At least 70% fertilization in controls **2.** The dilution water and the effluent egg blanks have essentially no eggs with fertilization membranes. **3.** Minimum significant difference (MSD) of <25% **4.** Final sperm stock concentration must be ≤33,600,000 sperm/mL AND one of the following conditions: **a.** Trial fertilization used – final sperm stock must not exceed double the target density selected that would provide 70-<100% fertilization without oversperming **b.** Sperm/egg ratio kept at ≤500:1 (without trial fertilization) and confirmation of sperm stock ≤5,600,000 sperm/mL **c.** Use any reasonable sperm stock density and run two extra sets of controls (high and low density). The high density control (0.200 mL of sperm stock) must have at least 5% higher fertilization than the low density control (0.050 mL of sperm stock)
WET Tests	
Larval Fish 7-Day Chronic Survival and Growth Test Topsmelt (*Atherinops affinis*) (EPA/600/R-95/136 – SOP No. TOX002.005)	**1.** Survival in the controls ≥ 80% **2.** 0.85 mg average weight of control larvae (9 days old)
Larval Fish 7-Day Chronic Survival and Growth Test Inland Silverside (*Menidia beryllina*) (EPA-821-R-02-014 – SOP No. TOX012.06)	**1.** Survival in the controls ≥ 80% **2.** 0.50 mg average weight of control larvae (7 days old)
Mysid Shrimp 7-Day Larval Survival, Growth, and Fecundity Test (EPA/821-R-02-14 – SOP No. TOX014B.02)	**1.** Survival in the controls ≥ 80% **2.** 0.20 mg average weight of control larvae (7 days old) **3.** MSD for growth ≤ 37% **4.** Egg production in ≥ 50% of females
Echinoderm Larval Survival and Development Test (EPA/600/R-95/136 – SOP No. TOX043.06)	**1.** Normal development in the controls ≥ 80% **2.** MSD for growth < 25%

Notes:

EPA = U.S. Environmental Protection Agency
mL = milliliter
MSD = minimum significant difference
SPP = suspended particulate phase
SOP = standard operating procedure

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2.7 Documents and Records

2.7.1 Responsibilities

The M-I SWACO NPDES Compliance Supervisor or designee establishes and maintains the central files for project records, training records, qualified subcontractor files, and other important documentation. Each M-I SWACO NPDES Compliance Specialist is responsible for assembling supporting documentation generated in his or her task and forwarding the documentation to the Shell Environmental Compliance Engineer and the M-I SWACO NPDES Compliance Supervisor at the end of the season. In addition, the M-I SWACO NPDES Compliance Specialist is responsible for providing the onsite laboratory reports to the permittee.

The M-I SWACO NPDES Compliance Supervisor and other signatories shall approve revisions of controlled documents such as the QAPP and associated SOPs and LWIs. The M-I SWACO NPDES Compliance Supervisor is responsible for determining, through consultation with technical staff, the activities that require SOPs and LWIs, and for working with the appropriate technical experts to develop the written procedures. The M-I SWACO NPDES Compliance Supervisor is responsible for obtaining technical review and approval of SOPs and LWIs, for maintaining control of QAPP and revisions, and for maintaining an up-to-date distribution list of written procedures.

M-I SWACO NPDES Compliance Specialists are responsible for performing tasks in accordance with applicable written procedures, except as explicitly directed by the relevant contract or Health and Safety policy. M-I SWACO NPDES Compliance Specialists are responsible for maintaining copies of applicable written procedures at the rig compliance laboratory locations, performing the requirements defined in by the written procedures, maintaining documentation as required by the written procedures, and notifying management of deviations from the written procedures. M-I SWACO NPDES Compliance Specialists are also responsible for assisting the Shell Environmental Compliance Engineer and/or the M-I SWACO NPDES Compliance Supervisor in designing accurate and practical written procedures, and in keeping the written procedures up-to-date.

2.7.2 Field and Laboratory Records

M-I SWACO shall retain copies of observations, calculations and derived data, calibration records, and a copy of the test report in accordance with the BMP and permit requirements. Records that are generated or stored by computers shall have hard copy or write-protected backup copies. Field notebooks shall be assigned an identification number and each page of the notebook shall be sequentially numbered. The M-I SWACO NPDES Compliance Supervisor shall maintain completed notebooks, hard copy electronic records, write-protected backup records, and test reports. Records that are expected to be generated to document compliance with drilling fluid and effluent monitoring requirements are provided in Table 2-36.

The records shall include the identity of the personnel involved in sampling, sample receipt, preparation calibration, and testing. Data, except those generated by automatic data collection systems, shall be recorded directly, promptly, and legibly in permanent ink.

Changes to records shall be signed or initialed by responsible staff. Entries shall not be obliterated by methods such as erasures, overwritten files, or markings. Corrections to recordkeeping errors shall be

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made by one line marked through the error. The individual making the corrections shall sign (or initial) and date the correction. This requirement also applies to electronically maintained records.

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Table 2-36 General Permit Compliance Records by Location

Record	Shell Quality Assurance/ Central Project Files	Compliance Specialist Notebook	M-I SWACO Area Office
Quality Assurance Project Plan (QAPP)	Original	Copy[2]	
Audit Reports	Original	Copy	
HSE Manual	Copy	Copy[2]	
Material Safety Data Sheet (MSDS)	Copy	Copy[2]	
SOPs and LWIs	Copy	Copy[2]	Original
Personnel Qualifications	Copy		Original
Training Documentation Form	Copy		Original
Demonstration of Capability	Copy	Copy	Original
Computer Program Verification	Original		
Equipment Manuals	Original	Copy	
Reference Material Certificates	Original	Copy	
Equipment Log	Original	Copy	
Field Notebook	Archive Copies[3]	Copy	
Daily Activity Report	Archive Copies[3]	Copy[2]	
Equipment calibration forms	Archive Copies[3]	Copy	
Sample chain-of-custody forms	Archive Copies	Copy	
Analytical reports from rig compliance laboratory	Archive Copies[3,4,5]	Copy	
Fixed analytical laboratory data packages	Archive Copies[3,4,5]		
Certification of Compliance – mercury and cadmium content in Barite	Archive Copies[3]	Copy	
Drilling Fluid Inventory	Archive Copies[3]		
Monthly Discharge Flow Volume Report	Archive Copies[3]		
Static Sheen Results	Archive Copies[3]	Copy	

Notes:

[1] Certified copy. *(continued on next page)*

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[2] Electronic copy.

[3] Rig compliance laboratory records will be retained in accordance with the permit requirements. Fixed laboratories shall maintain originals of their analytical data in accordance with their internal QA programs.

[4] Including QC results, if applicable.

[5] Copy of CoC document, with recipient's signature at sample transfer to be filed. Sample transfer is defined as the point at which the sample is delivered to any third party.

[6] Originals provided to permittee at required frequency (e.g., at end of drilling season).

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2.7.2.1 Deviations

Deviations from SOPs, if any, shall be documented on Corrective Action Report Forms (Table 2-37).

CAR No. _____

Date: _____

Table 2-37 Service Quality Non-Conformance Report

To:	From:
Findings or Deviations	
Answer Due Date	Signature
Review and Comments	
Date	Signature
Review and Comments	Follow up Actions

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2.8 Reporting

2.8.1 Notifications

If any of the discharge limits are exceeded or if other suspected BMP Plan noncompliance or modification occurs, Shell Compliance Duty Officer (907-830-7435) must be called immediately. Noncompliance of the NPDES GP that may endanger health or the environment (if any) must be reported to the EPA by telephone within 24 hours from the time of occurrence. This includes any unanticipated bypass or upset that exceeds discharge limitations in the permit or any violation of maximum daily discharge limitations for any of the pollutants listed in Part 1 of the GP requiring 24-hr reporting.

If NPDES GP noncompliance occurs, the Shell Environmental Compliance Engineer will complete and file the necessary reports to the EPA in accordance with permit requirements.

2.8.2 Discharge Monitoring Reports

All discharge monitoring results and effluent sampling will be summarized in the Discharge Monitoring Report (DMR) form EPA No. 3320-1 or equivalent. Monitoring data and other reports will be submitted electronically using NetDMR (http://www.epa.gov/netdmr). DMRs will be submitted to EPA no later than the 20th of the month following the completed reporting period.

All permit records are submitted to Shell. Annual sampling results will be reported on the January DMR. All records of monitoring information shall be retained at least 5 years from the date of the sample, measurement, report, or application.

The permittee must ensure that records of monitoring information include:

- The date, exact place, time of sampling or measurements, and the name(s) of the individual(s) who performed the sampling or measurements;
- The date(s) analyses were performed and the names of the individual(s) who performed the analyses;
- The analytical techniques or methods used; and
- The results of such analyses.

Noncompliance reporting that is not required to be reported within 24 hours is to be included with the Discharge Monitoring Reports and submitted monthly. Shell is to preserve all reports and records for a period of at least five years from the date of sample, measurement, report, or application, or for the term of this permit, whichever is longer.

2.8.3 TAH/TAqH Reporting

Total aromatic hydrocarbons, or TAH, is determined by summing the results of EPA Method 602 (plus xylenes) or EPA Method 624 to quantify monoaromatic hydrocarbons. Total aqueous hydrocarbons, or TAqH, are determined by summing the results for TAH and EPA Method 610 or EPA Method 625 (to quantify PAHs listed in EPA Method 610). For samples where one or more analytes has a detectable result, TAH and TAqH will be presented as the sum of the detectable results. Detectable results include results that are reported as estimated (e.g., J-flagged) because the result is greater than the detection limit

and less than the reporting limit. If all results for the sample are nondetect, one-half the reporting limits for all nondetect results will be summed.

2.9 Data Reporting

A group of samples submitted to the subcontract laboratory at the same time and included on the same CoC form will be considered a sample delivery group. The results for this sample delivery group will be reported as one analytical data package. The analytical data package must contain adequate information to verify the quality of the data and be presented in a clear and concise manner. Data packages must include, at minimum, the following elements:

- Cover sheet, which identifies the project;
- Table of contents;
- Case narrative, which documents all discrepancies with the data contained in the report, including (but not limited to) sample receipt, holding time(s), documentation of QC discrepancies and corrective action, matrix interferences;
- Preparation and analytical methods used;
- Sample identification;
- Analytical results, including detection limits, reporting limits, and dilution factors;
- Laboratory qualifiers;
- Date(s)/time(s) of collection, receipt, preparation, analysis;
- Sample receipt and management records; and
- QA/QC sample results and supporting information.

The toxicity testing laboratory is required to report results that include all information recommended by the test protocols for quality assurance review and data validation, as follows:

- Test methods used for toxicity testing and statistical analyses;
- Source of testing water including a description of any pretreatment, and results demonstrating survival and growth of test organism in test water;
- Source, history, and age of test organisms and if appropriate culturing information, or collection information. Records should include information regarding taxonomic identification of test organisms;
- Source of food composition and procedures used to prepare and dispense food to test chambers;
- Description of experimental design including test setup, test monitoring, and test termination. Water quality and observation records should be summarized and included in report;
- Methods used for physical and chemical characterization of test matrices;
- A table of biological data for each sample, including negative and positive control information;
- Methods used for statistical analysis;

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- A description of any deviations from the methodology or problems with the process and procedures of analyses;
- Original data sheets for water quality, survival, growth, abnormalities, reference toxicant, and statistics as applicable by test protocol;
- Chain-of-custody records; and
- References and literature.

2.10 Data Review and Qualification

All analytical data that the laboratory generates shall be verified before submittal to the permittee. This internal data review process, which is multi-tiered, shall include all aspects of data generation, reduction, and QC assessment. All definitive data shall be reviewed first by the analyst, and then by the supervisor of the respective analytical section using the same criteria. Elements for review or verification at each level must include, but are not limited to, the following:

- Sample receipt procedures and conditions;
- Sample preparation;
- Appropriate analytical SOPs and methodologies;
- Accuracy and completeness of analytical results;
- Correct interpretation of all raw data, including all manual integrations;
- Appropriate application of QC samples and compliance with established control limits;
- Verification of data transfers;
- Documentation completeness; and
- Accuracy and completeness of data deliverables (hard copy and electronic).

2.10.1 Laboratory Data Evaluation

The calibration, QC, corrective actions, and flagging requirements will be performed in accordance with the laboratory QA Manual and the analytical methods. The laboratory shall apply data qualifiers as part of its internal validation activities. Flagging criteria apply when acceptance criteria are not met and when corrective actions are not successful or not performed. The supervisor of the respective analytical sections shall review the data qualifiers.

The laboratory's QA section shall perform internal review prior to issuance of the final data packages. The laboratory project representative shall complete a final review on all of the completed data packages and issue the final reports.

2.10.2 Data Review and Verification

Staff reviewing permit compliance data will conduct a QA/QC review and assessment of laboratory data deliverables, including an evaluation of:

- The information provided on the analytical data sheets, including QC sample results;

- The laboratory case narrative and any flags that the laboratory applied as part of its data validation and usability assessment;
- The sample collection documentation, including CoC records; and
- Field laboratory data sheets and supporting documentation.

This review will also verify the accuracy and completeness of field activities, including the adherence to the procedures as described in the cited SOPs and the specified analytical methods. In general, analytical data that have been reviewed by the laboratory QA section and permit compliance staff will be considered acceptable for use, including results that were flagged as part of the laboratory QA review and validation. Laboratory flags and case narratives will be reviewed as part of the data quality review and assessment to confirm that the quality of the data is acceptable for its intended purpose.

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3.0 Environmental Monitoring Program

3.1 Introduction and Overview

In the management of its activities, Shell has contracted with Olgoonik Fairweather, LLC for the implementation of the Environmental Monitoring Program (EMP) portion of its exploration program in the Chukchi Sea. This section addresses the GP compliance tasks that will be performed by Olgoonik Fairweather LLC under contract to Shell during offshore drilling exploration.

Table 3-1 defines the roles and responsibilities of the EMP implementation team. The specific details of responsibilities may change during project execution. Changes will be documented and communicated to the project team to ensure that project personnel are aware of appropriate points of contact and to avoid any gaps in responsibilities

Table 3-1 Roles and Responsibilities of the EMP Implementation Team

Role	Responsibilities
Shell Science Lead (SL)	• Technical input on and review of project scope and equipment specifications • Participation in identification of risks, opportunities, and hazards
Shell Project Lead (PL)	• Ultimate program responsibility and management • Project execution • Permitting • Accounting • Stakeholder communication
Shell Representative (Shell Rep)	• Offshore based single point accountability for operations and oversees the projects assets and contractors in regards to program delivery, health, safety, security and environment (HSSE), reputation, stakeholder and regulatory compliance requirements established for the project • Coordination of operational implementation in the field • Sample shipping logistics • Day to day communication with Shell
Vessel Master (VM)	• Is the primary operator of the vessel • In United States (US) waters, must comply with US Coast Guard (USCG), state, and local regulations • Is responsible for all aspects of boating operations, regardless of any senior personnel present on the boat. These responsibilities include, but are not limited to: o Safety of the vessel and all persons on board o Safe transport of the vessel to and from her berth, if applicable o The safe operation of all shipboard equipment o Ensuring that all required operational and safety equipment is on board and that crew and passengers know the location and how to operate safety/survival equipment o Report all accidents, incidents, boardings, citations, safety concerns according to USCG regulations
Olgoonik Fairweather Project Manager (PM)	• Planning, coordination, and execution of the program as defined by Shell • Oversight of project assets and subcontractors
Olgoonik Fairweather Lead Scientist (LS)	• Development of EMP design and quality assurance project plan (QAPP) • Coordination and approval of study reports • Oversight of field and analytical activities

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Role	Responsibilities
Sr Marine Technician (MT)	• Field Instrument/Equipment Calibration, Maintenance, and Operation • Works with VM to determine if conditions are acceptable for deployment of sampling tools • Oversight and coordination (e.g., between VM and CS) of deployment and retrieval of sampling gear
Principle Investigator(s) (PI)	• Onshore based lead scientist for particular scientific field or subcontractor • Contribute expertise to development of sampling design, EMP, QAPP, and final reports. • Plan field sampling and execute post-field processing activities • Review laboratory data for reasonableness and usability • Communication of quality assurance (QA) and quality control (QC) requirements to project personnel
Chief Scientist (CS)	• Offshore based overall field scientific lead • Ensures field sampling procedures comply with QAPP and Standard Operating Procedures (SOPs) are being followed • Documents sample collection and processing activities, and initiates sample custody and/or additional sampling • Maintain custody of field records during ship-board activities • Review field records at the end of each sampling day
Field Team(s) (FT)	• Collect the samples after the sampling equipment is retrieved • Take control of the collected samples once determined to be acceptable and maintain custody of samples collected for chemical analysis • Process and preserve samples according to the QAPP, including all field documentation • Prepare and ship samples under custody to the appropriate analytical laboratories
Laboratory Manager(s) (Lab Manager)	• Conduct all sample analysis, reporting, analytical activities, as well as all activities related to sample custody records and processing in accordance with their contracts and the QAPP and SOPs • Review/QC data, assign laboratory qualifiers, and implement corrective action • Ensure independent QA oversight • Communicate any issues that could affect sample integrity, data quality, or schedule to contracting Laboratory QA/QC Officer • Perform internal verification and validation of all reported data • Submit data packages and electronic data deliverables (EDDs) that conform to QAPP requirements
Laboratory QA/QC Officer (Lab QA/QC)	• Review analytical data to verify that QAPP and SOP requirements were achieved • Ensure data are traceable to raw data, calculations are accurate • Ensure data qualifiers are applied to any data that do not meet the QAPP measurement quality objectives (MQO) requirements • Report the results of audit to management

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3.2 Data Quality Objectives

The data quality objectives of the EMP are to address the following NPDES GP requirements:

1) Complete an initial site assessment, including a physical sea bottom survey, to ensure the exploratory facility is not located or anchored in a sensitive biological area or habitat;
2) Evaluate water-quality characteristics of the receiving water and potential effects of the specified discharges;
3) Evaluate sediment characteristics of the seafloor and potential effects of the discharges on the sediment characteristics;
4) Evaluate potential effects to the benthic community structure due to deposition of Discharge 001 (water-based drilling fluids and drill cuttings) and Discharge 013 (muds, cuttings, cement at the seafloor), which includes both spatial and temporal changes in community diversity and abundance; and
5) Evaluate the plume(s) in the vicinity of the discharges

3.3 EMP Technical Approach: (Phase I through IV)

The technical approach for the EMP requires collection of data during four different phases shown in Table 3-2. The types of analysis for the four phases are shown in Table 3-3. The sampling design described in Section 3.5 will describe each of the four phases and how they meet the DQOs of the EMP.

The sampling design is based on and intended to meet the data quality objectives (DQOs) of the EMP.

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Table 3-2 Summary of Field Sampling for the Four Monitoring Phases

EMP Component	Matrix	Permit Reference	Existing Baseline Data (Phase I replacement) (pre-drill baseline)[1]	Phase II (during)	Phase III (post-drill)	Phase IV (no later than 15 months post drill)
Physical Oceanography/ Meteorology	Physical Characteristics (water)	II.A.f.2.	(X)[2]			
	Plume Monitoring (water)	II.A.j.4		X		
Biology	Benthic Community Structure (benthos/epibenthos)	II.A.f.4 II.A.i.2	X			X
	Physical Sea Bottom Survey	II.A.f.1. II.A.h.1. II.A.i.1	X		X	X
Chemistry	Water-based Drilling Fluids/Drill Cuttings	II.A.j.1		X		
	Sediment Characteristics (surface sediment)	II.A.j.2	X		X	X
	Benthic Community Bioaccumulation Monitoring (tissue)	II.A.j.3	X		X	X
Chemistry (continued)	Receiving Water Chemistry	II.A.f.3.	(X)[3]	X		
	Plume Monitoring (water)	II.A.j.4		X		

Notes:

[1] As indicated in the EMP, Appendix A, Phase I data includes historical data within the previous 5 years, sampling completed in 2012, 2013, additional samples of opportunity , and samples that may be collected concurrent with Phase II.

[2] Water column physical characteristics have historically been collected and additional data may be collected during Phase II.

[3] Reference samples will be collected outside the plume during Phase II as the most representative baseline (Phase I) data during drilling.

3.3.1 Phase I Assessments

The Phase I data collected from the previous 5 years was compiled and analyzed to determine the variability within and among the data sets from the same region and to establish whether historical data from a larger geographical area may be predictive of current baseline data at site-specific locations. The results demonstrate that baseline data are available and are sufficient for replacement of Phase I permit requirements; therefore, Phase I sampling requirements are not addressed in this QAPP. The results of this analysis are presented in the EMP Appendix A.

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Table 3-3 Summary of Analytical Parameters by Matrix

Parameter	Water Total	Water Particulate Phase	Water Dissolved Phase	Sediment	Tissue	Drilling Fluids/Cuttings[1]
Volatile Organic Carbon (VOC)/ Total Aromatic Hydrocarbons (TAH)[2,3]	(Phase II)[4]					Phase II
Saturated Hydrocarbons (SHC)	(Phase II)			Phase III Phase IV	Phase III Phase IV	Phase II
Total Petroleum Hydrocarbons (TPH)	(Phase II)			Phase III Phase IV	Phase III Phase IV	Phase II
Polycyclic Aromatic Hydrocarbons (PAH)	(Phase II)			Phase III Phase IV	Phase III Phase IV	Phase II
Petroleum Biomarkers				Phase III Phase IV	Phase III Phase IV	Phase II
Percent Lipid					Phase III Phase IV	
Particulate Organic Carbon (POC)	(Phase II)					
Total suspended solids (TSS)	(Phase II)					
Grain Size				Phase III Phase IV		
Total organic carbon (TOC)				Phase III Phase IV		
Sediment profile imaging (Sea Bottom) or ROV camera survey				Phase III Phase IV		
Benthic Community Structure (benthos/epibenthos)				Phase IV		
Aluminum (Al)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Antimony (Sb)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II

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Parameter	Water Total	Water Particulate Phase	Water Dissolved Phase	Sediment	Tissue	Drilling Fluids/Cuttings[1]
Arsenic (As)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Barium (Ba)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Beryllium (Be)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Cadmium (Cd)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Chromium (Cr)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Copper (Cu)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Iron (Fe)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Lead (Pb)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Manganese (Mn)				Phase III Phase IV	Phase III Phase IV	Phase II
Mercury (Hg)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Methyl mercury			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Nickel (Ni)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Selenium (Se)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Silver (Ag)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Tin (Sn)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II

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Parameter	Water Total	Water Particulate Phase	Water Dissolved Phase	Sediment	Tissue	Drilling Fluids/Cuttings[1]
Titanium (Ti)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Thallium (Tl)			Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Vanadium (V)				Phase III Phase IV	Phase III Phase IV	Phase II
Zinc (Zn)		Phase II	Phase II	Phase III Phase IV	Phase III Phase IV	Phase II
Total metals		6	19	19[5]	19[5]	19[5]

Notes:

[1] Analyses for total recoverable [metals] concentrations must be conducted and reported for each metal using EPA methods. The results must be reported in "mg/kg of whole mud (dry weight) and moisture content (percent by weight) of the original drilling fluid sample."

[2] TAH "as determined by EPA Method 602 (plus xylenes)".

[3] Total Aqueous hydrocarbons (TAqH) is the sum of TAH + PAH.

[4] As indicated in the EMP, Appendix A, Phase I data includes historical data, sampling completed in 2012, 2013 additional samples of opportunity, and samples that may be collected concurrent with Phase II.

[5] All required metals (Table A, NPDES GP) are included in the 19 metals. Manganese and vanadium are also included in sediments, muds and cuttings, and tissue sample analyses bringing the total metals for those matrices and phases to 21.

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3.3.2 Phase II Plume Monitoring and Observations

The objective of the plume-monitoring component is to measure metals, organics, turbidity and total suspended solids throughout the water column during periods of maximum discharge of water-based drilling fluid and drill-cuttings (Discharge 001). Additionally, the objective is to focus characterization efforts on areas of expected deposition of water-based drilling fluid and drill-cuttings based on model predictions. Plume monitoring will also serve as a check/verification of modeled effluent behavior.

Phase II plume monitoring will be conducted from a vessel configured to conduct environmental monitoring. Safety, operational and navigational issues could limit the ability to delineate plumes in the immediate vicinity of the drilling operations. Within these logistical constraints, efforts will be made to safely locate and sample the plume(s) during the drilling process. In order to assess maximum discharge of metals, hydrocarbons, turbidity, and total suspended solids, two primary time periods will be targeted.

(1) Drilling of the largest casing interval after the BOP stack is set; this time period represents the expected maximum discharge rate over the longest time interval of water-based drilling fluids and drill-cuttings during the drilling process.

(2) During and/or immediately following bulk drilling fluid discharge; this discharge represents a period when only water based drilling fluid (with some finer entrained drill cuttings) is discharged and total suspended solids could be higher due to the small particle size of the material (barite and bentonite).

In the event that sampling during these time points cannot be conducted in the field, secondary options are presented in the EMP. During the two discharge events listed above, up to seven sampling stations will be targeted for sample collection (Figure 3-1). A total of up to 70 water samples will be targeted for collection plus 6 samples of drill cuttings and drilling mud will be collected during Phase II (Table 3-4). The direction of the predominant currents will be measured with the ADCP system and will be used to inform actual sampling stations during field activities.

The data collected during the Phase II monitoring will be used to assess the location of the plume(s), to refine model inputs, and to help inform the Phase III and IV monitoring efforts.

Figure 3-1 Phase II Water Sampling Stations

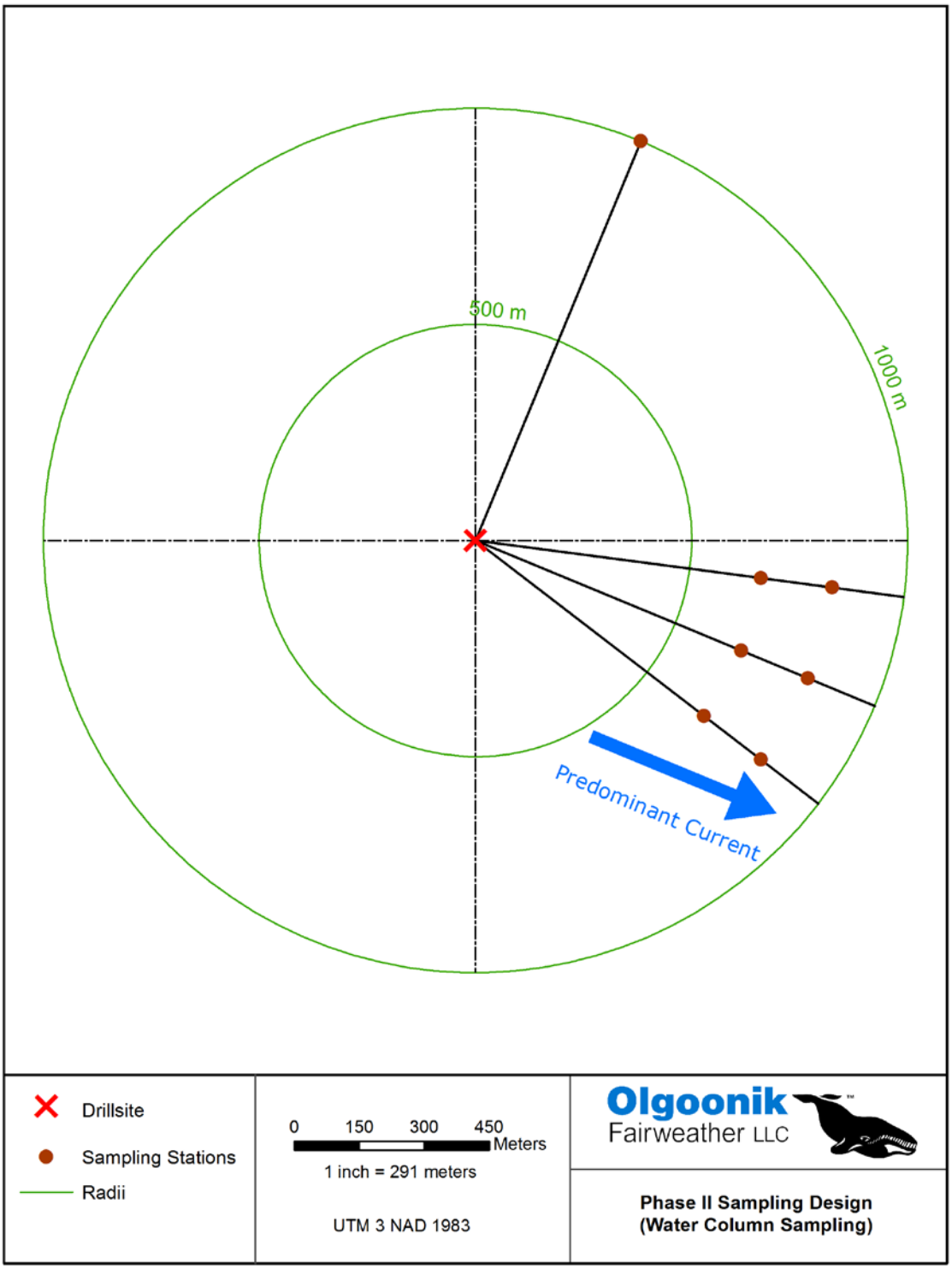

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Table 3-4 Number of Samples Slated for Collection During Phase II

Sampling Water Depth[1]	Transect Type	Number of Samples (Estimated)			
		Phase – Largest Casing Interval	Phase – Bulk Drilling Fluids[2]	Other Intervals[4]	Total Number of Samples
1 m below surface	Plume	6	6	---	12
	Reference	1	1	---	2
10 m below surface	Plume	6	6	---	12
	Reference	1	1	---	2
20 m below surface	Plume	6	6	---	12
	Reference	1	1	---	2
30 m below surface	Plume	6	6	---	12
	Reference	1	1	---	2
2 m above bottom	Plume	6	6	---	12
	Reference	1	1	---	2
Drill-Cuttings	Drilling Rig	2	0[3]	2	4
Drilling Fluid	Drilling Rig	2	2	2	6
Subtotal		**Up to 39**	**Up to 37**	**Up to 4**	**Up to 80**

Notes:

[1] Sampling water depth may vary depending on in-field measurements of turbidity during plume monitoring, weather conditions, or operational parameters.

[2] If bulk discharge event occurs.

[3] No separate drill cuttings samples will be collected because they are not present at significant concentrations in the bulk drilling fluids.

[4] Drill-cuttings and drilling fluids will be collected at each interval beyond the top hole as specified in the Drilling Fluids Plan.

3.3.3 Phase III Assessment

Phase III incorporates the post drill sampling immediately (as soon as practicable) following cessation of drilling at a well site. In the event that unforeseen circumstances occur preventing the environmental sampling of data immediately after drilling, the EPA will be notified immediately to determine the next course of action. A four-transect design (N, E, S, and W) off-set 22.5 degrees in line with the annual mean current direction, in conjunction with four different radii at 100 m, 250 m, 500 m, and 1000 m from the drill site location, will be used (Figure 3-2; Table 3-5).

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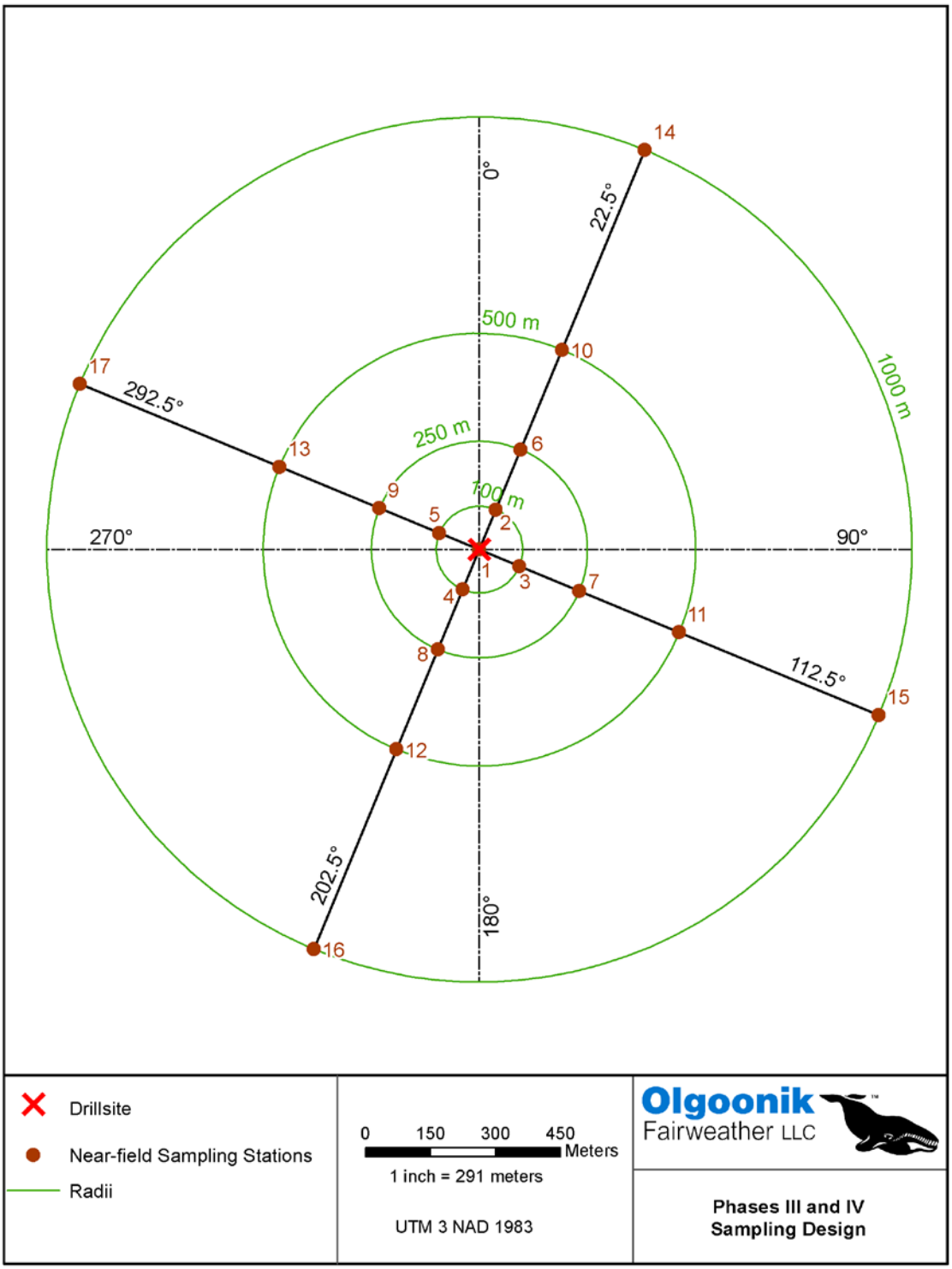
Figure 3-2 Phase III and IV Sampling Stations



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These transect orientations may be modified in the field, depending on observations made during the field effort (e.g., the Phase II ADCP data indicate a different trajectory for the predominant downstream current direction), and/or sediment profile imaging or ROV survey, and sediment grab samples will be used to also help determine the depositional area of the muds and cuttings.

Table 3-5 Summary of Near-Field and Far Field[1] Phase III and IV Samples Slated for Collection

Discipline	Number of Sample Design Near-field stations	Number of Far-Field[1] stations	Number of samples
Sediment Profile Imagery	Up to 17	Up to 2-4	Up to 19-21[2]
Benthic ecology (Phase IV only)	Up to 17	Up to 2-4	Up to 57-63 (3 reps, possibly 5 reps, depending on field conditions and operational limitations)
Chemistry—sediments	Up to 17	Up to 2-4	Up to 19-21
Chemistry—biota (clams)	Up to 4	Up to 1-2	Up to 5-6
Chemistry—biota (amphipods)	Up to 4	Up to 1-2	Up to 5-6

Notes:

[1] Far-field samples will be collected at 2-4 stations contemporaneous with the near-field stations. Far-field stations will be consistent with a subset of stations from the CSESP, where possible.

[2] Multiple photographs will be taken at each station (plan-view and cross-sectional) to ensure at least one high-quality photograph per station.

3.3.4 Phase IV Assessment

The sampling that occurs for the Phase IV monitoring must follow the same sampling design as for the Phase III sampling, as per the NPDES permit (Figure 3-2; Table 3-5). Sediment profile imaging (or similar technology), sediment chemistry, and tissue chemistry sample collection for Phase IV, if possible (e.g., if organisms are present for sample collection for chemical analysis), will be identical to the Phase III sampling design. Benthic community structure will be added for the Phase IV assessment to measure and assess any potential long term impacts to the benthic community as a result of the exploratory drilling operations. Three to five replicate sediment samples for identification and enumeration of benthic infauna will be collected from up to17 near-field stations and 2-4 far-field reference locations.

3.4 Field Sampling

3.4.1 Sampling Methods

The following section summarizes the field sampling procedures that will be used to implement the environmental monitoring plan. Details are provided in field SOPs provided in Appendix B.

3.4.1.1 Navigation

The Shell vessel navigation system will be used to track vessel and sampling locations during environmental monitoring if the accuracy of the system meets the EMP requirements (±10 m). If the

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vessel navigation system cannot meet this level of accuracy, an auxiliary system will be provided to define sampling locations. The location of each sample is marked as a waypoint when the FT communicates to the VM that a sample is about to be collected. The milestone number is documented in the field log by the FT and the pre-assigned sample identification (ID) number is recorded with the waypoint number, creating a link between each sample and the station coordinates.

3.4.1.2 Acoustic Doppler Current Profiler (ADCP)

The subsurface ADCP or similar technology will be positioned no more than 2000 m from the drill site. The data on current speed and direction will be relayed in real-time or near-real time fashion from the units to the vessel so that the field team can use them to maximize the effectiveness of the Phase II plume-sampling component. The current data will provide an estimate of the trajectory of the plume in the field. Discrete water samples then will be collected from the sampling stations.

3.4.1.3 Conductivity, Temperature, and Depth (CTD)

A Sea-Bird Electronics, Inc., SBE19*plus* V2 (or similar) CTD profiler with (at a minimum) a turbidity sensor (e.g., optical backscatter [OBS]) and a transmissometer will provide real-time multi-dimensional data on suspended solids to optimize water-sampling locations. The CTD may be mounted on the rosette water sampler and may be used to collect hydrographic data at each water station, depending on the apparatus used to collect water samples (e.g., a pumping system may be used instead of a rosette).

Sensor measurements will be collected during the downcast from near surface (approximately 1 meter) to within approximately 2 m of the sea floor at each station. Salinity and density (as sigma-t) will be calculated using Sea-Bird software from the conductivity, temperature and depth data. Navigational position and time will be recorded concurrently with the hydrocast measurements. CTD profiles and the presence of suspended solids detected by the turbidity sensor(s) and transmissometer will indicate the presence of the plume in addition to ADCP water column velocity data and real-time weather conditions. Water samples will be collected during the upcast or downcast (depending on rosette type, to minimize sample contamination) within the depth zones indicating plume characteristics. CTD operation will follow the guidance of Battelle SOP 5-275 *At-Sea Collection of Hydrographic Data Using CTD and Rosette System.*

3.4.1.4 Water Samples

A total of up to 70 water samples representing the five different sampling depths in the water-column will be targeted for collection during Phase II using a multiple bottle rosette sampler (e.g., example pictured in Figure 3-3) or similar system. Samples will be collected in the rosette using GO-FLO or Niskin bottles. The sampling layout is depicted in Figure 3-1. Table 3-4 summarizes the sampling design. Sampling collection using the rosette sampler will follow the guidance of Battelle SOP 5-275 *At-Sea Collection of Hydrographic Data Using CTD and Rosette System.*

Transect locations will maximize the plume coverage in response to discharge and plume dynamics. The general procedure during each sampling event is proposed as follows:

1) The field team will use the real-time or near-real time ADCP data (if available) along with recent water column velocity data to determine the current direction and speed near the drilling operation.

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2) Prior to initiation of plume sampling, whole-water samples and CTD/turbidity data will be collected along the reference transect located at least 1,000 m away and perpendicular to the up-current end of the downstream plume transect (Figure 3-1).

3) The plume-monitoring transect will be conducted across the plume (laterally) at intervals from as close to the release point as it is safe to occupy and down-current until evidence of the plume (e.g., turbidity measurements above background levels) disappears (Figure 3-1). ADCP data will be used to help locate the plume vertically and horizontally based on water volume velocity data. Modifications to the transect scheme may be made at the discretion of the CS in response to conditions encountered in the field.

4) Additional vessel transects along the plume (axially) may be made at the discretion of the field leader to help locate plume boundaries, pending vessel logistical considerations and priorities in the field.

5) Water samples will be collected with a sampling rosette within the plume at up to six stations along three transects (two stations per transect) oriented in the direction of the predominant current at each of five depths. The main plume transect will be positioned along the primary current direction. Two additional transects will be set at approximately ±10-15 degrees from the primary current direction (Figure 3-1). All plume-transect sampling stations will be attempted within 1000 m from the drilling location, with the near-field stations being as close to the discharge as logistically possible. The sampling vessel will be equipped with a laboratory supporting on-board water filtration for collection of water samples for analysis of dissolved metals. Water samples will be filtered as soon as practical on-board the vessel laboratory.

6) ADCP and CTD data will be used in an adaptive manner to optimize the location and depth for discrete water sample collection to capture the densest portion of the plume when possible. General target sample depths are approximately 1 m (near-surface), 10 m, 20 m, 30 m below the surface of the water, and 2 m above the sea bottom.

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Figure 3-3 Rosette Water Sampler with Go-Flow Bottles and CTD Sensors

3.4.1.5 Muds and Cuttings

Two samples of used water-based drilling fluids and two samples of drill cuttings will be collected during drilling of the largest casing interval (after the blowout preventer (BOP) is set), and two samples of used water-based drilling fluids will be collected during bulk-mud discharge (if applicable), in Phase II of the monitoring program, for a total of 6 samples (Table 3-4). In addition, Permit No.: AKG-28-8100 Section II.A.13.j.1 requires metals analysis of each drilling fluid system. Different weight drilling systems are utilized in each drilling interval as described in the Drilling Fluids Plan. A sample from each drilling interval beyond the top hole (as specified in the Drilling Fluids Plan, i.e., 17 ½", 12 ¼", and 8 ½" hole sizes) will be collected and analyzed for the metals identified in Table A of Permit No.: AKG-28-8100 (Additional 4 samples shown in column termed "Other Intervals" in Table 3-4). Drilling-fluid compositions and monitoring records will be obtained from Daily Mud Report and Well Mud recap (done at end of well) to the degree possible.

The water-based drilling fluids and drill cuttings will be collected by personnel on the Shell drilling rig (e.g., M-I SWACO NPDES Compliance Specialists) and placed in clean glass jars. Sample handling and analysis will be performed in accordance with Section 3.5.2. Samples will be frozen immediately to ensure preservation.

3.4.1.6 Sediment Samples

Sediments will be sampled at up to 17 near-field stations and up to two to four far-field stations during Phases III and IV with a double van Veen grab sampler (Table 3-5). Samples will be collected from the top 2 cm (i.e., the surficial layer) of sediment. Depending on sediment observations from van Veen grab collections, gravity-core samples also may be collected in the field to obtain truly undisturbed cross-sectional samples of the sediment layer and to provide information on the depth/thickness of solids deposition caused by Discharges 001 and 013. The thickness of any overlying materials (water-based drilling fluids and drill cuttings) will be estimated by the CS or appropriate FT lead at the time of sample collection and recorded as part of the field documentation efforts.

Van Veen Grab Sampler

The double van Veen grab sampler is designed to be deployed from a vessel equipped with a power winch and A-frame or boom system and to collect undisturbed surface sediment samples to a maximal depth of approximately 15 cm. The double van Veen grab sampler enables chemistry and benthic infaunal samples to be co-collected from the same deployment. Fairweather Science (FWS) SOP 06 *Surface Sediment Sampling Using a Modified van Veen Grab Sampler* describes the operation of the grab sampler. Based on the sediment surface, the penetration depth of the grab sampler will be modified for softer or denser sediments. Weight will be added for dense, stiff sediment or removed for soft sediments. A sheet of material (e.g., plastic, plywood) may be fastened to the grab frame to slow the descent rate into the sediment to prevent over-penetration. In addition, the grab sampler can be used in a variety of sea conditions.

Upon return of the grab sampler to the deck of the vessel, the sampler will be placed on a table to enable sediment collection from the buckets. The hinged access doors on the top of the sampler are opened to determine whether the sample is acceptable for collecting material for analysis. The discipline specific FT lead makes this determination. An acceptable sample collection is one that displays the following characteristics:

- Sampler is not overfilled with sediment, the jaws are fully closed and the top of the sediment is below the level of the open doors.
- Overlying water must be present and must not be excessively turbid.
- No significant leakage of water from the sampler of the bucket used for collecting a sample, unless sediment is very coarse grained (e.g., gravel or coarse sand).
- The sampler is at least 80% full of sediment or the desired penetration has been achieved.
- The sediment is level on at least one side.

Overlying water with no excessive turbidity indicates that the sediment sample is undisturbed and that surface sediments remain intact (i.e., there was no significant leakage of water and, hence, fine sediment

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remains in the sampler). If unacceptable, the grab sampler will be redeployed at a slightly different location to avoid disturbance of the sediment layer from previous unacceptable grabs and drops of unwanted sediment.

If the grab sample is acceptable then samples will be collected according to the following steps:

1) Remove overlying water from the grab by siphoning or pumping through a pre-cleaned Teflon tube. (See Section 3.5.1.11 for equipment decontamination procedures).
2) Collect samples through the access doors into stainless steel bowls and/or clean glass sample jar, avoiding sediment that contacts the metal sides of the grab.
3) Collect the top 2 cm of sediment in the grab using a Kynar®-coated (or equivalent) scoop pre-marked with a 2-cm marking. This surficial sediment layer represents recent accumulation (in this case, potentially drilling muds and cuttings discharged as a result of the drilling operations) and incorporates the primary biogenic zone.

Once the top 2 cm have been removed from the grab into the homogenizing container, it will be gently but thoroughly mixed to a homogeneous color and consistency and subsampled into the appropriate containers for analysis of organics, metals, grain size, and total organic carbon (See Table 3-6). Any rocks, large shells, or rock debris will be removed from the sample.

Gravity Core

If collected, the sediment-core samples would be obtained most likely in the immediate vicinity of the drilling location and at the stations located within the 100 m and 250 m concentric radii from the drill site. If evidence exists in the field of muds or cuttings thicker than expected beyond the 100 m radii, additional core samples may be taken. Core samples would be collected only if/when sediment samples collected by grab or other data indicated substantial thickness of drilling muds or cuttings. Decisions regarding additional coring will be made at the discretion of the CS. Any sediment core samples would be in addition to the scope of the surface sediment sampling described earlier (e.g., in Table 3-5).

Battelle SOP 5-342 *Collecting Sediment Cores with a Piston Push/Hammer Corer* describes the operation of the gravity core. After collection, the core liner with sediment will be removed from the outer core tube. Following penetration and removal, the core will be examined for integrity and volume. The core is acceptable if the appropriate length is collected and the sediment recovered is relatively undisturbed throughout its depth. Disturbed cores will be rejected; a new core liner installed; and the station will be resampled. If not immediately processed, the core liner will be sealed at each end with electrical tape or an equivalent system (e.g. cable tie) and stored upright in ice for up to 6 hours prior to processing. Pre-cleaned core liners will be used to collect each sample; a new liner will be used for each station. The cores must be chilled (Table 3-6) and stored upright until processing.

Core sample processing will be conducted in the field on the vessel as follows:

1) Place the core liner with sediment onto a clean working surface.
2) Cut the core liner length-wise with a clean stainless steel knife.
3) Split the core length-wise using a clean stainless steel knife and/or spatula.

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4) Gently open the two halves and remove any pieces of liner material in contact with sediment.

5) Collect the sediment in each core segment using a pre-cleaned stainless steel or Kynar® (or equivalent) coated spoon or spatula into a clean stainless steel bowl. The length of each core segment will based on observed horizons identified by the CS. It is anticipated that segment thicknesses could range from 2 – 10 cm. Core samples may also instead be extruded from the core barrel in the field (rather than splitting the core).

6) Gently but thoroughly mix the sediment to a homogeneous color and consistency and subsampled into the appropriate containers for analysis of organics, metals, grain size, and total organic carbon (See Table 3-6).

Table 3-6 Sample Containers, Sample Sizes, Preservative Requirements, and Holding Times for Sediments, Tissue, and Water

Compound Class	Minimum Sample Size	Container[1]	Preservation	Holding Time[2]
Sediment/Water-Based Drilling Fluids/Cuttings				
Hydrocarbons[3]	8oz	Glass with Teflon lid	Frozen –20°C	1 year to extraction/40 days to analysis
VOC, muds and cuttings only	10g	Glass with Teflon lid	Cool <6ºC	14 days
Metals	2oz	Glass with Teflon lid	Frozen –20°C	1 year
Mercury	4oz (1/2 filled)	Glass with Teflon lid	Frozen –20°C	1 year
Methylmercury	4oz (1/2 filled)	Glass with Teflon	Frozen –20°C	1 year
TOC	2 oz	Glass	Cool <6°C, above freezing	6 months
Grain Size	4 qt	Plastic bag	Cool <6°C, above freezing	6 months
Benthic Animals	60ml	Whirl-Pak® plastic bags, various sizes	Buffered Formalin	1 month
Benthic Animals	60 mL	Glass jars	Transfer from formalin to ethanol	Indefinite
Tissue				
Hydrocarbons	8oz	Glass with Teflon	Frozen –20 °C	1 year to extraction/40 days to analysis
Metals	2oz	Glass with Teflon	Frozen –20 °C	1 year
Mercury	4oz	Glass with Teflon	Frozen –20 °C	1 year
Methylmercury	4oz	Glass with Teflon	Frozen –20 °C	1 year
Water				

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Compound Class	Minimum Sample Size	Container[1]	Preservation	Holding Time[2]
VOC[4]	3 x 40 ml vials	Glass with Teflon-lined lid	No head space. Acidified to a pH ≤2 with hydrochloric acid, above freezing	14 days
Hydrocarbons[5]	2 1 L	Glass with Teflon-lined lid	Cool <6°C, above freezing	14 days to extraction/40 days to analysis
Metals (dissolved)	1L	HDPE	Acidified to a pH ≤2 with Nitric Acid	6 months
Metals (particulate filter)	1 mg on filter[6]	Plastic petri dish; double Ziploc bags	Dry	6 months
Mercury	0.5 L	Teflon	Acidified to a pH <2 with Hydrochloric Acid	90 days
Methylmercury	0.5 L	Glass with Teflon-lined lid	Acidified to a pH <2 with Hydrochloric Acid	180 days
POC	1	Glass Fiber filter in plastic petri dish	Frozen	1 year
TSS	1	Polycarbonate Filter	Frozen in sealed petri dish	6 months

Notes:

[1] Container Types: G = Glass/Teflon-lined lid; P=Plastic SPEX.

[2] "x" days/"y" days = maximum days from sampling to extraction/maximum days from extraction to analysis.

[3] PAH, SHC/TPH, petroleum biomarkers.

[4] Three 40-ml VOC vials w/ Teflon® septum and Zero head space.

[5] PAH, SHC/TPH.

[6] Volume filtered on 0.47-mm, 0.4-µm pore size polycarbonate filter based on the TSS concentration is typically 0.5 – 2.0 L.

3.4.1.7 Sediment Samples - Benthic Ecology Samples

Infaunal and epifaunal benthic invertebrates will be sampled with a double van Veen grab sampler at up to 17 near field stations and up to two to four far-field stations. Three to five replicate grab samples will be collected at each station (Table 3-5, Figure 3-2). Sediment samples for benthic ecology will be collected using the same protocol as for sediment samples collected for environmental chemistry. FWS SOP-06 *Surface Sediment Sampling Using a Modified van Veen Grab Sampler* describes the operation of the grab sampler. Photographs of the surface of the grab samples will be taken to compare to Plan View photographs taken during the SPI or ROV program.

Following the completion of the sediment sample collection at a particular station, a clean plastic bin will be placed underneath each side of the non-disturbed (no sediment removal for chemistry) grab samplers to empty the bucket with the remaining sediment for biota sorting (both for benthic ecology and biota chemical analysis samples). Samples will be rinsed using a 1.0-mm mesh stainless steel screen with water pressure sufficient enough to remove sediments but weak enough to minimize damage to the animals to isolate biological material (a stacked 0.5 mm sieve may also be placed beneath the 1.0 mm sieve to

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capture smaller organisms). The bucket that was used for sediment chemistry sampling may be used to obtain additional tissue mass for chemistry samples. Rinse water will be seawater filtered at least as low as the smallest sieve to remove any indigenous pelagic organisms that may be introduced into the benthic sample. Biological and sediment residues will be carefully removed from the sieve so that all visible animals, sediment particles, and fragments will be removed. The biological and sediment residue remaining in each sieve will be placed into separate pre-labeled plastic jars or Whirl-Pak® bags, preserved in 10% formalin buffered with borax or equivalent and securely stored for shipment to the laboratory. The sieving and sample preservation procedures for benthic ecology samples are described in Environ SOPSAM043.01 *Benthic Sample Collection and Processing for Shell Baseline Environmental Sampling Program – Chukchi Sea*.

3.4.1.8 Tissue Samples for Chemistry

Collection of biota samples (clams and amphipods) will be attempted at up to four (4) near-field stations and up to one to two additional reference stations each during both Phases III and IV (Table 3-5). Target locations for biota sampling will be stations 3, 7, 11, and 15 but the actual locations will be determined in the field based on the availability of clams and amphipods. Due to natural patchiness of the biota, it is unlikely that biota samples will be able to be collected at all specific proposed stations. Biota samples for tissue analysis will be collected by using a combination of a clam rake and double van Veen grab sampler at the same station for clams and by using baited modified minnow traps for amphipods. Previous efforts at collecting bivalves and other benthic organisms in the Chukchi Sea (J. Hardin, Battelle, San Diego, CA, pers. obs.) indicated that clams are not obtained in numbers large enough for tissue volumes required for chemical analyses with the double van Veen grab sampler. Alternatively, the use of a clam rake towed for a few minutes often provides adequate numbers of bivalves. Because sample size is important for chemical analysis in order to have enough sample mass to meet target detection limits for all analyses, the use of the clam rake is warranted for bivalve collection. FWS SOP-12 *Benthic Tissue Sampling for Chemical Analysis Using a Dredge/Rake* and FWS SOP-13 *Amphipod Sampling for Chemical Analysis Using a Modified Minnow Trap* describe the collection procedures.

Following tissue sample collection, the catch is brought aboard and emptied into large plastic tubs. The contents of the tubs then are sorted by trained personnel using clean-gloved hands. Target bivalve species include *Astarte* spp. and *Macoma* spp. If numbers are sufficient to provide the required tissue mass, one and the same species will be collected at each station; however, if this is not possible then it is more important to collect sufficient tissue mass for the analyses. Preliminary identification of the bivalve species will be determined in the field and verified by taxonomic identification. Collected bivalves are placed in a clean sieve and gently rinsed with seawater to remove external debris prior to being counted and photographed. Clams will be placed into pre-cleaned glass containers, and frozen. Similarly, amphipods will be gently rinsed free of any visually adhered materials and will be placed into pre-cleaned glass containers and frozen (Table 3-6). Chemical analysis will be conducted separately on whole body clams and on whole body amphipods (i.e., the organisms will not be dissected prior to chemical analysis).

3.4.1.9 Sediment Profile Imaging

Plan-view digital photographs of the seafloor and sediment profile images (cross-sectional digital photographs of the sediment-water interface) will be obtained using SPI technology at up to 17 near field stations per drill site and up to two to four reference stations. Images will be assessed to characterize

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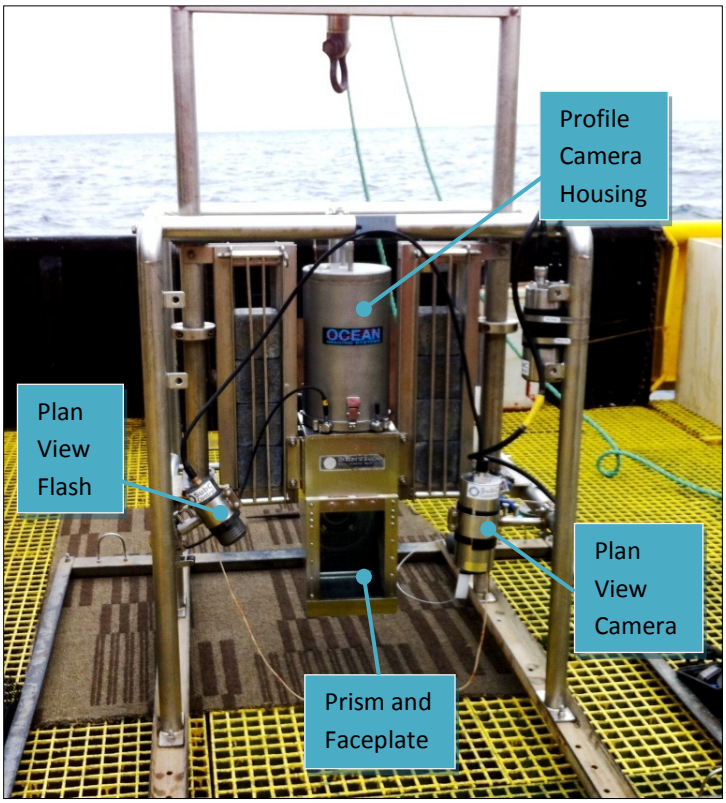
baseline seafloor conditions. Operation of the SPI will follow the guidance of NewFields SAM044.01 *Sediment Profile Imaging (SPI) and Plan View (PV) Photography Collection.*

The SPI system consists of a camera enclosed in a waterproof, pressure-resistant housing, a 45° prism that penetrates the sediment, and a mirror that reflects an image of the sediment profile through the camera lens to a digital camera (Figure 3-4). The camera frame also supports a downward-looking camera to view the surface of the seafloor. These images are viewed onboard the vessel after the system has been retrieved and permit the scientific crew to view conditions of the seafloor and the ability to provide near real-time guidance of sampling activities and strategy. The two independent camera systems are triggered by mechanical switches. The plan-view camera is triggered first at a pre-determined elevation above the sea bottom. When the system hits the sea bottom and the prism begins to penetrate the sediment, the SPI camera switch initiates time-delay electronics that trigger the camera to collect an image when the prism has completed penetration (~15 seconds). At each station, the camera is lowered to the seafloor a minimum of three times to ensure that replicate images suitable for analysis are obtained. At any station where difficulties are encountered, additional camera drops can be made. The date, time, station, water depth, photo number, and estimated prism penetration are recorded in a field log, with each drop of the camera also logged. The digital images are transferred to an onboard computer after each station for back-up. Images are transferred to a third location (e.g., an on-vessel external hard-drive) at least 1 time/day.

Figure 3-4 Sediment Profile Imaging and Plan View Camera Systems



Alternatively, photography of the ocean floor can be accomplished by the use of an ROV equipped with a downward-facing central digital camera with two flashes on both sides. The device may be placed in a

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sealed titanium housing with a window allowing for wide angle photographs, depending on water depth and pressure considerations. The distance between the sea floor bottom and the camera is known through the use of an altimeter on the ROV. If used, the ROV system provides both digital photographs and video recordings of sea bed characteristics and the epibenthic infaunal community structure only.

The ROV is powered through a hydraulic propulsion system which is supplied with power from the surface vessel through an electrical control umbilical. The ROV is deployed from the vessel or rig via an electrical winch that is attached to a tether management system (TMS). Once deployed, the TMS is suspended vertically below the surface vessel at an altitude above the seafloor of approximately 10 m. The ROV is then able to exit the TMS and make access to the seafloor to conduct its visual inspection of the sampling stations. Dependent on the ROV system in use, a lateral offset (maximum flying distance) between the ROV and TMS would be no greater than 100m. A video system consisting of multiple cameras allows the operators to direct the ROV and manipulate both mechanical arms and other systems. The ROV will be lowered to within 1-2 meter of the seafloor or to a distance such that the sea bed is not disturbed by the ROV propulsion system. Photographs and video footage at each sampling site will be collected for approximately 30 minutes at each sampling station visited or as required by the onboard science crew. Imagery is recorded in real time onto digital hard drives that are located onboard the vessel or rig. The digital images are transferred to an external hard drive after each station for back-up. Images are transferred to a third location (e.g., an on-vessel external hard-drive) at least 1 time/day.

3.4.1.10 Contamination Prevention

Extreme care will be taken to avoid cross-contamination of sampling equipment and samples during collection and processing. FWS SOP-04 *Decontamination of Equipment – Sediment* and FWS SOP-07 *Decontamination of Tissue Sampling Equipment* describe general procedures for decontamination of field sampling equipment. Other procedures include the following:

- Avoid contact with hydrocarbon sources and any possible metals contamination during the collection of samples.
- Use only clean stainless steel and/or plastic equipment and utensils for sample collection.
- Decontaminate all components of the sampling equipment prior to use.
- Store sampling equipment away from grease drips from winches and winch wire, diesel smoke, and other potential airborne contaminants (e.g., smoking) when it is not being used to sample.
- Process samples in a clean area outside the influence of gasoline or diesel engines fumes and exhaust gases.

Rigorous decontamination procedures will be used to ensure that sampling equipment is clean and comply with the QAAP. The CS will be responsible for assuring SOPs are followed.

To the extent possible, non-contaminating, pre-cleaned materials (glass, stainless steel, Teflon™) will be used for sample collection. When this is not possible (steel equipment, core liners), the sampling equipment will be cleaned as described below; 1% Alconox® solution (or equivalent) will be used as soap. The use of solvents as part of the equipment decontamination process will be avoided, if possible, to limit the generation of solvent waste. However, if any oily sheen is noted or if oil contamination is

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suspected, then the affected equipment will be rinsed three times with methanol. The methanol must be completely evaporated prior to use of the equipment.

- Any utensils that will contact samples for chemical analysis (e.g., stainless steel spoons or spatulas, screens and forceps) will be scrubbed with soap and water and rinsed three times with deionized water.
- During the sample collection and processing, field personnel will wear nitrile (or equivalent) gloves that will be changed between stations.
- Sample coring equipment will be cleaned prior to use and between samples. A new core liner will be used for each station. Other components of the core will be thoroughly scrubbed with a stiff brush and soapy water and thoroughly rinsed with site water at the beginning of each day and between each sampling station.
- All sources of contamination (airborne sources, fingers, unclean equipment) should be avoided.
- The core liner will be sealed at both ends to contain the sediment and keep contamination out. The core sample will not be removed from the liner until it is ready for processing under controlled conditions.

3.4.2 Sample Handling and Custody

The procedures described in this section are designed to ensure that sample integrity and custody are maintained at all times. If the sampling labeling, preservation, holding times, or custody requirements are not met, Shell will be notified. The laboratory may proceed with testing with client approval and the data will be qualified by including a discussion of any protocol deviations in the analysis report. If deemed feasible, the client may elect to provide a replacement sample for the out-of-specification sample and the initial sample rejected for further testing.

3.4.2.1 Sample Labeling

A unique sample identification (ID) number must be assigned to each sample. The sample ID must be documented by the CS in the field logbook and communicated to the ship navigation system so that sample location and collection information are definitively linked to a specific sample container. The sample ID is a concatenation of the sample year, station ID, replicate number, and sample type in the form as shown in Table 3-7. For example, a sample collected from Burger A drill site, station BA001a, which is replicate 3 for benthic ecology would be identified as: 14-BA001a-03-BE.

Table 3-7 Sample Identification Scheme

Character Position in Sample ID	Description	Example
1,2	Year	14 (i.e., 2014)
3-8	Station ID	BA001a
9,10	Replicate Number	03
11,12	Sample Type	BE
Sample Identification Code Definitions		
Codes for Sample Types	QC Sample Types	Visit Number

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AC = Amphipod chemistry BC = Bivalve chemistry BE = Benthic ecology CC = Cuttings chemistry DC = Drilling fluid chemistry GS = Grain size LC = Water (liquids) chemistry PV = Sediment Profile Imaging plan view camera photograph SC = Sediment chemistry SP = Sediment Profile Imaging photograph TOC = Total organic carbon	EB = Equipment blank FB = Field blank TB = Trip blank (VOCs)	A unique sample ID if re-sampling is needed. The default is 01, and is incremented each time a sample is recollected.

Each sample must be labeled with the unique sample identification number as soon as it is containerized. Sample labels must provide sufficient detail to uniquely identify each sediment sample and allow tracking to field activities. An example label is shown in Figure 3-5.

Figure 3-5 Example Sample Label

Project Chukchi Sea_____ **Sample ID**_____

Collection Date (YYMMDD)_____Time (2400) **Station ID**_____

Matrix (Circle one): Sediment / Water / Tissue / Drilling Fluid / Cuttings

Water Type (Circle one): Whole / Particulates / Dissolved

Tissue Type (Circle one or enter other): Macoma / Astarte / Other: _____

Analysis Type: Organics Metals POC TSS TOC Grain size Biology

Preservation (Circle): Chill Freeze Acidify Formalin None Other_____

Sample Collector Initials:_____

Container ___ of ___ (*e.g.*, 1 of 2, 2 of 2) if the sample is contained in more than one container.

3.4.2.2 Sample Preservation and Packing for Shipment

Once samples are transferred to sample containers for analysis, they will be preserved as specified in Table 3-6 and maintained at the required temperature/conditions until packaged for shipment to the laboratories. FWS SOP-03 *Sample Packaging and Shipping* describes these procedures. Every effort will be made to deliver the samples to the analytical laboratories in a timely manner to meet the sample holding times.

To prepare samples for shipment,

- Protect the sample jars with shock insulation such as bubble wrap. The cooler should have insulation placed on the bottom of the cooler and the samples should be wrapped in insulation if breakable or crushable containers are used.

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- For coolers containing samples for chemical analysis, add gel ice or cubitainers of frozen water to achieve the proper temperature and to ensure that the samples stay at a constant temperature for their entire trip.
- Pack samples tightly so that they cannot move freely in the cooler; they must be secure.
- An upper weight limit of 50 pounds per cooler is suggested.
- For coolers containing samples for chemical analysis, place a temperature blank container in each cooler.
- Record the contents of the coolers and list them on the chain-of-custody (CoC) forms.
- Affix two CoC seals on each cooler with samples. Packing and labeling will conform to IATA regulations for the sample preservative.

3.4.2.3 Holding Times

Sample holding conditions and holding times are defined in Table 3-6. Holding times are determined from the time of sample collection. Documentation must be sufficient to track sample holding, processing, and analysis times to ensure that holding times are met. Samples must be held in a controlled area with limited access. Some data results with short holding times may be flagged due to the logistics of transporting the samples from the remote site to the analytical laboratories. These data are expected to still be usable within the confines of the data quality objectives presented in this QAPP.

Field samples collected for organics or metals analysis will be held for six months after delivery of final data; sample extracts and digestates will be held for one month. Disposal records for unextracted samples, extracted samples, sample containers, and sample extracts must be sufficient to provide tracking from collection, through laboratory receipt, to sample disposal.

3.4.2.4 Chain-of-Custody Records

Sample custody records are the administrative records associated with the physical possession and/or storage history of each individual sample from sample collection to the final analytical result and sample disposal. FWS SOP-02 *Sample Custody* and FWS SOP-03 *Sample Packaging and Shipping* describe these procedures. Sample custody will be initiated by the sample collection records that identify for each unique sample identification number the date, time, collection location, and collector.

During the survey, samples will be in the custody of the FT(s), who will store samples securely under the preservation requirements defined in Table 3-6. When samples are packaged for shipment to the analytical laboratories, the FT(s) will verify that each sample is recorded on the appropriate custody form. Each sample custody form will be signed by the FT(s), relinquishing the samples once he/she has verified that the custody form is accurate; that all samples present in the shipping container are listed on the form, and that the sample descriptions, requested analytical methods, and sampling dates are accurate. The sample custody form provides a record of the samples collected and analyses requested. The custody form should be sealed in a plastic bag and taped to the inside lid of the cooler. The original sample custody forms accompany the samples; the shipper will keep a copy.

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If more than one cooler is sent in one shipment to the laboratory, then each cooler will contain a separate custody record for the samples in that cooler. In addition, the outside of the coolers will be marked to indicate the number of coolers in the shipment (*e.g.*, 1 of 2, 2 of 2). Two custody seals will be placed on each cooler lid on opposite sides and will be signed and dated.

Upon receipt at the designated laboratory, sample custody forms will be signed by the person receiving the samples once that person has verified that all samples identified on the custody forms are present in the shipping container. Any discrepancies will be noted on the form (in addition to any internal laboratory documentation policy) and the sample receiver will immediately contact the Lab Manager to report missing, broken, or compromised samples.

Each analytical laboratory must have a formal, documented system designed to provide sufficient information to reconstruct the history of each sample, including preparation of sampling containers, sample collection and shipment, receipt, distribution, analysis, storage or disposal, and data reporting within the laboratory. Laboratory documentation must provide a record of custody for each sample throughout processing, analysis, and disposal.

Field custody of electronic data, including all data on navigation, CTD, dissolved oxygen, ADCP acoustic backscatter, and optical-backscatter turbidity will be the responsibility of the field survey's CS. The field custody of the electronic data consists of creating external hard-drive backups of all electronic data generated each day. The label on the backup media will include a survey ID, date, and name of person creating the backup files. The data will be transferred to a software system capable of physical oceanographic processing upon completion of the survey.

3.4.2.5 Sample Archiving

Samples must be archived under the conditions specified in Table 3-6 until the final analytical data have been received, reviewed, and approved by the scientific field specific PI.

3.4.3 Field Instrument/Equipment Calibration, Maintenance, and Operation

Field equipment must be tested, maintained, and calibrated according to SOPs and the manufacturers' instructions prior to use in the field to avoid breakdowns that could impact schedule or loss of data. The instruction manuals and SOPs must be available for all field equipment so that trouble-shooting and routine repairs can be conducted in the field. Spare parts recommended by the manufacturer should be stocked on the vessel. Major maintenance should be documented in the field logbook. Maintenance must be documented to track instrument performance or problems. Documentation should include the name of the person performing the maintenance, date maintenance was performed, a description of the maintenance activity, and (if the maintenance was performed in response to a specific instrument performance problem) the result of re-testing to demonstrate that the instrument performance had been returned to acceptable standards prior to re-use. Calibration, maintenance, testing, and inspection requirements for field equipment are defined in Table 3-8 and is the responsibility of the MT.

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3.5 Laboratory Analysis

3.5.1 Analytical Methods

Laboratory-specific detection limits and reporting limits have been evaluated against the EMP objectives, and have been deemed sufficient for their intended uses.

Table 3-9 summarizes the methods that will be utilized by matrix and parameter. As noted on the table, more than one method may be used for trace metals. Analytical laboratory SOPs and QA/QC data will be provided to the EPA upon request.

Table 3-8 Field Equipment Calibration, Maintenance, Testing, and Inspection

Equipment	Activity	Frequency
Rosette- Sea Bird SBE19plus CTD and sensors (or equivalent)[1]	Calibration	Prior to survey
	Maintenance	Prior to survey; as necessary
	Testing	After any modifications/changes
	Inspection	Visual inspection before and after each cast, data reviewed after each cast
GPS[2]	Calibration	NA
	Maintenance	As necessary (GPS systems do not require scheduled maintenance)
	Testing	As necessary (GPS systems do not require scheduled testing)
	Inspection	As per Ship's standard procedures, data accuracy will be continuous during critical navigation operations
Sediment Profile Imaging System[3]	Calibration	Prior to survey
	Maintenance	Prior to survey; as necessary during survey (no scheduled maintenance)
	Testing	After each cast
	Inspection	Visual inspection before and after each cast; photographs reviewed after each cast

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Equipment	Activity	Frequency
ADCP	Calibration	Prior to survey
	Maintenance	Prior to survey and deployment
	Testing	Prior to survey and deployment.
ROV System	Maintenance	Ongoing as required for continued operations

Notes:

[1] Samples/direct measurements collected: Conductivity/salinity, temperature, depth, fluorescence, turbidity, transmissivity.

[2] Samples/direct measurements collected: Location of sampling equipment at time of sample or direct measurement. [3] Samples/direct measurements collected: Sediment profile images, sediment characterization.

Table 3-9 Laboratory Analytical Methods

Compound Class (Units)	Method (EPA Citation)	Possible Laboratory[1]	Laboratory SOP Analysis Method
Water			
VOCs	EPA 8260[2]	Battelle – Norwell	5-245 GC/MS purge and trap
PAH	EPA 8270	Battelle – Norwell	5-157 GC/MS
SHC/TPH	EPA 8015 (Mod)	Battelle – Norwell	5-202 GC/FID
Metals (Dissolved): As, Be, Cd, Cr, Cu, Ni, Pb, Sb, Se, Tl, Zn	EPA 6010C	Florida Institute of Technology	FIT-0004 Series ICP-MS[3]
Metals (Dissolved): Ag, Al, Ba , Fe, Mn, Sn, Ti, V	EPA 6010C	Florida Institute of Technology	FIT-0005 Series ICP-MS[4]
Mercury (Dissolved)	EPA 1631E	Battelle - Sequim	MSL-I-013 CVAF
Methylmercury (Dissolved)	EPA 1630	Battelle - Sequim	MSL-I-014 CVAF
Metals (Particulates): Ba, Cr, Zn	EPA 6020A	Florida Institute of Technology	FIT-6005 Series ICP-MS[5]
Metals (Particulates): Al, Fe	EPA 7000B	Florida Institute of Technology	FIT-6003 Series FAAS
POC (Particulates)	Modified EPA 415.1	Florida Institute of Technology	FIT-2015 TOC TOC Analyzer
TSS (Particulates)	ASTM 3977-07, Method B or EPA 160.2	Florida Institute of Technology	FIT-TSS Analytical Balance
Sediment and Drilling Fluids/Cuttings			
VOCs, muds and cuttings only	EPA 8260	Battelle - Norwell	5-245 GC/MS purge and trap
PAH	EPA 8270	Battelle - Norwell	5-157 GC/MS
Petroleum Biomarkers	EPA 8270 (Mod)	Battelle - Norwell	5-157

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Compound Class (Units)	Method (EPA Citation)	Possible Laboratory[1]	Laboratory SOP Analysis Method
			GC/MS
SHC/TPH	EPA 8015 (Mod)	Battelle - Norwell	5-202 GC/FID
Metal: Ag, As, Ba, Be, Cd, Ni, Pb, Sb, Se, Sn, Tl, V	EPA 6020A	Florida Institute of Technology	FIT-2012 Series ICP-MS
Metals: Al, Cr, Cu, Fe, Mn, Ti, Zn	EPA 7000B	Florida Institute of Technology	FIT-2010 Series FAAS
Total Mercury	EPA 7473	Battelle - Sequim	FIT-2014 CVAAS
Methylmercury	EPA 1630M	Battelle - Sequim	MSL-I-015 CVAF
TOC	Modified EPA 415.1	Florida Institute of Technology	FIT-2015 TOC Analyzer
Grain Size	NA	Florida Institute of Technology	FIT-2010-GS
Tissue			
PAH/Petroleum	EPA 8270	Battelle - Norwell	5-157 GC/MS
Petroleum Biomarkers	EPA 8270 (Mod)	Battelle - Norwell	5-157 GC/MS
SHC/TPH	EPA 8015 (Mod)	Battelle - Norwell	5-202 GC/FID
Metal: Ag, As, Ba, Be, Cd, Ni, Pb, Sb, Se, Sn, Tl, V	EPA 6020A	Florida Institute of Technology	FIT-1012 Series ICP-MS
Metals: Al, Cr, Cu, Fe, Mn, Ti, Zn	EPA 7000B	Florida Institute of Technology	FIT-1013 Series FAAS
Total Mercury	EPA 7473	Battelle - Sequim	MSL-I-034 CVAAS
Methylmercury	EPA 1630M	Battelle - Sequim	MSL-I-015 CVAF
Percent Lipid	NA	Battelle - Norwell	SOP 5-190 Gravimetric

Notes:

[1]Possible analytical laboratories are listed. Laboratories may change prior to project implementation and may include ALS Global (Kelso, WA for hydrocarbons, TOC, Grain Size analysis) and Brooks Rand Laboratory (Seattle, WA for metals analysis) or other contract analytical laboratory/laboratorie(s).

[2] VOC compounds are the Method 602 compounds plus o, m, p-xylene.

[3] Inductively-coupled plasma-mass spectrometer (ICP-MS) following preconcentration using method of Nakashima et al. (1988).

[4] Inductively-coupled plasma-mass spectrometer (ICP-MS) following preconcentration using a SeaFast system (Hathorne et al. 2012).

[5] USEPA SW-846 Method Series 6**010C** with analysis by inductively coupled plasma/mass spectrometry (ICP-MS) according to FIT SOP 6005.

3.5.2 Samples for Metals Analysis

Samples of drill cuttings, mud samples, water, sediments, and tissues will be analyzed for a suite of metals. The analyses will be conducted following protocols that have been developed specifically for reliable trace-level analysis of the target metals in complex marine environmental samples. The analytical

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protocols have been used extensively for baseline characterization and monitoring the potential impact of offshore oil and gas activities in Alaska, including in the CSESP, Chukchi Offshore Monitoring In Drilling Area – Chemistry and Benthos (COMIDA-CAB), Arctic Nearshore Impact Monitoring In Development Area (ANIMIDA), and Continuation of Arctic Nearshore Impact Monitoring In Development Area (cANIMIDA) programs. Table 3-10 lists the metals that will be analyzed for each phase of the EMP and the applicable analytical instrument.

3.5.2.1 Metals in Water (Phase II) Samples

Dissolved metals in water samples collected during drilling activities (Phase II) will be analyzed for the list of 19 metals (Table 3-10). The dissolved-phase metal samples will be pre-concentrated prior to analysis. Particulate-bound metals in water samples collected during the plume-monitoring component in Phase II will be analyzed for 6 metals (Table 3-10) known to be indicators of particles derived from drilling muds and cuttings; the suspended particles will be air-dried (at 50% humidity). The samples will be analyzed by flame atomic absorption spectrometry (FAAS), graphite furnace atomic absorption spectrometry (GFAAS; with Zeeman or Continuum background correction [ZGFAAS]), or inductively coupled plasma/mass spectrometry (ICP/MS) for determination of the different metals. Mercury concentrations will be measured by Cold Vapor Atomic Fluorescence **(**CVAF). These methods are based on USEPA methods described for Series 7000B (FAAS and GFAAS), Series 1630 and 1631E (CVAF), and Series 6010C (ICP/MS), with optimization to address the required detection limits and the sample matrices.

3.5.2.2 Metals in Sediment (Phases II, III, and IV) Samples

Sediment samples and muds and cuttings samples will be analyzed for a suite of 19 metals (see Table 3-10). The well-mixed mud/cuttings and sediment will be freeze-dried and then totally digested in Teflon® beakers using concentrated high-purity hydrofluoric acid (HF), nitric acid (HNO_3) and perchloric acid ($HClO_4$). The liquid-phase and clear samples will be diluted with distilled deionized water (DDW) prior to analysis. The dissolved-phase water samples will be concentrated prior to analysis. Sediment samples to be analyzed for mercury will be digested by heating with HNO_3 and sulfuric acid (H_2SO_4). The samples will be analyzed by FAAS, CVAF, cold vapor atomic absorption spectrometry (CVAAS), or ICP/MS for determination of the different metals. Total mercury concentrations will be measured by CVAAS and methylmercury by CVAF. These methods are based on USEPA methods described for Series 7000 (FAAS and ICP-MS), Series 7473 (CVAAS), Series 1630M (CVAF), and Series 6010A (ICP/MS, with optimization to address the required detection limits and the sample matrices). Analyses of muds and cuttings for concentrations of total recoverable metals will be conducted following EPA methods, and results will be reported in mg/kg of whole mud (dry weight) and moisture content (percent by weight) of the original drilling-fluid sample. Laboratory limits of quantitation (LOQ) and limits of detection (LOD) are defined in Table 3-10.

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Table 3-10 Metals and Analytical Instruments for Each Phase of Environmental Monitoring with Laboratory Limits[1]

Analyte	Analytical Instrument[2]	Dissolved Metals Water		Particulate Metals Water		Sediment &Drilling Mud/Cuttings		Tissue	
		LOQ (µg/L)	LOD (µg/L)[3]	LOQ	LOD	LOQ (µg/g)	LOD (µg/g)	LOQ (µg/g)	LOD (µg/g)
Aluminum (Al)	ICP-MS, FAAS	TBD[4]	TBD	0.15 %	0.03 %	0.015%	0.003%	10	2
Antimony (Sb)	ICP-MS	0.010	0.002	0.2 µg/g	0.04 µg/g	0.005	0.0014	0.005	0.001
Arsenic (As)	ICP-MS	0.02	0.004	-	-	0.10	0.02	0.06	0.012
Barium (Ba)	ICP-MS	0.075	0.015	9 µg/g	1.8 µg/g	0.05	0.01	0.05	0.01
Beryllium (Be)	ICP-MS	TBD	TBD	-	-	0.005	0.001	0.005	0.001
Cadmium (Cd)	ICP-MS	0.0015	0.0003	-	-	0.025	0.005	0.005	0.001
Chromium (Cr)	ICP-MS, FAAS	0.015	0.003	1.5 µg/g	0.3 µg/g	8	1.6	0.05	0.01
Copper (Cu)	FAAS	0.010	0.002	-	-	8.5	1.7	0.01	0.002
Iron (Fe)	FAAS	TBD	TBD	0.15 %	0.03 %	0.05%	0.01%	12.5	2.5
Manganese (Mn)	ICP-MS, FAAS	0.02	0.004	-	-	15	3	5	1
Mercury (Hg), Total	CVAF (Dissolved) CVAAS (other)	0.5 ng/L	1 ng/L	-	-	5 ng/g	2 ng/g	5 ng/g	1.9 ng/g
Methylmercury (MeHg)	CVAF	0.05 ng/L	0.02 ng/L	-	-	0.05 ng/g	0.01 ng/g	5 ng/g	1.5 ng/g
Nickel (Ni)	ICP-MS	0.01	0.002	-	-	0.20	0.04	0.05	0.01
Lead (Pb)	ICP-MS	0.002	0.0004	-	-	0.10	0.02	0.015	0.003
Selenium (Se)	ICP-MS	0.075	0.015	-	-	0.1	0.02	0.075	0.015
Silver (Ag)	ICP-MS	0.003	0.0006	-	-	0.015	0.003	0.02	0.004
Tin (Sn)	ICP-MS	0.030	0.006	-	-	0.05	0.01	0.01	0.002
Titanium (Ti)	ICP-MS, FAAS	TBD	TBD	-	-	0.1%	0.02%	0.1%	0.02%
Thallium (Tl)	ICP-MS	0.35 ng/L	0.07 ng/L	-	-	0.015	0.003	0.005	0.001
Vanadium (V)	ICP-MS	-	-	-	-	0.01	0.01	0.05	0.01
Zinc (Zn)	ICP-MS, FAAS	0.015	0.003	3.5 µg/g	0.8 µg/g	2.5	0.5	2	0.4

Notes:

[1] Actual detection limits (e.g., LOD, MDL) may be higher or lower, depending on the specific analytical laboratory under contract at the time of project implementation.

[2] CVAAS = Cold Vapor Atomic Absorption Spectrometry; CVAF = Cold Vapor Atomic Fluorescence; FAAS = Flame Atomic Absorption Spectrometry; GFAAS = Graphite Furnace Atomic Absorption Spectrometry; and ICP/MS = Inductively Coupled Plasma/Mass Spectrometry.

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[3] Laboratory detection limits are verified and updated periodically according to FIT's quality system. Detection limits current at the time of analysis may vary from those defined in this QAPP. The actual laboratory LOQs and LODs will be reported with the laboratory data.

[4] To be determined (TBD). Laboratory LODs will be developed and reported with the study data.

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3.5.2.3 Metals in Tissue (Phases III and IV) Samples

Tissue samples will be analyzed for a suite of 19 metals (Table 3-10). Tissue samples for all metals, with the exception of Hg, will be freeze-dried and then digested by sequential addition of concentrated, high-purity nitric acid (HNO_3), hydrogen peroxide (H_2O_2), and hydrochloric acid (HCl). The solution will be diluted with distilled/deionized (DDW). Mercury analyses will be conducted following digestion with concentrated, high-purity HNO_3 and H_2SO_4

The samples will be analyzed by FAAS, CVAF, or ICP/MS for determination of the different metals. Mercury concentrations will be measured by CVAF and CVAAS. These methods are based on USEPA methods described for Series 7000B (FAAS), Series 1630M (CVAF), Series 7473 (CVAAS), and Series 6010C (ICP/MS, with optimization for particular sample matrices).

3.5.3 Samples for Hydrocarbon Analysis (Phases II, III, and IV)

Samples of drilling mud, cuttings, sediment, and tissues will be analyzed for a suite of PAH, petroleum biomarkers (sterane/triterpanes; St/Tr), TPH, and SHC compounds. Water samples collected during Phase II monitoring will be analyzed for VOCs, TPAH, SHC and TPH; but, petroleum biomarkers will not be measured in the water samples. The analyses will be conducted following SOPs (e.g., 5-157, see Table 3-9) that have been developed specifically for reliable trace-level analysis of the target parameters in complex marine environmental samples (e.g., Trefry et al. 2003 and 2012). The analytical protocols have been used extensively for baseline characterization and monitoring the potential impact of offshore oil and gas activities in Alaska, including in the CSESP, ANIMIDA, and cANIMIDA programs for hydrocarbon analysis.

The instrumental analysis will be conducted following methods that are modified from US EPA Methods 8015 (SHC) and 8270 (PAH and biomarkers), to obtain improved sensitivity and specificity, to include a number of additional key target parameters (e.g., alkyl PAHs and petroleum biomarkers), and to ensure that the analysis is appropriate for complex samples of drilling mud/cuttings, sediments, and biological tissues. The sample analyses are summarized below.

3.5.3.1 Hydrocarbons in Water Samples (Phase II)

Water samples will be extracted for PAH, SHC, TPH, and VOC compounds (Table 3-11, Table 3-12, and Table 3-13) following laboratory SOPs. Briefly, water samples will be prepared by fortifying a 1–2 L sample with surrogate internal standard (SIS) compounds, serially extracting the analytes of interest with dichloromethane (DCM), and preparing the samples for instrumental analysis. The sample extract will be dried and concentrated over anhydrous sodium sulfate and then will be purified with an alumina clean-up column. The extract will be concentrated and spiked with internal standards (IS) and split for instrumental analysis. One split will be submitted for SHC and TPH analysis by modified EPA Method 8015 by using gas chromatography with flame-ionization detection (GC/FID). The other split will be submitted for PAH analysis that uses a modified EPA Method 8270 gas chromatography/mass spectrometry (GC/MS) method with the detector operating in the selected ion-monitoring (SIM) mode. Target compounds will be quantified by using the method of internal standards using the SIS compounds resulting in surrogate recovery-corrected data generated to represent the native sample concentrations. Laboratory reporting limits (RL) and method detection limits (MDLs) are reported in Table 3-11, Table 3-12 and Table 3-13.

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Table 3-11 List of Polycyclic Aromatic Hydrocarbon and Alkyl PAH Target Analytes with Reporting Limits and Method Detection Limits[1]

Parameter	Whole Water (ng/L)		Sediment (ng/g dry)		Tissues (ng/g dry)	
	RL	MDL[2]	RL	MDL	RL	MDL
Naphthalene[3]	5.0	3.28	1.2	0.531	2.9	2.98
C1-Naphthalenes	5.0	3.28	1.2	0.531	2.9	2.98
C2-Naphthalenes	5.0	3.28	1.2	0.531	2.9	2.98
C3-Naphthalenes	5.0	3.28	1.2	0.531	2.9	2.98
C4-Naphthalenes	5.0	3.28	1.2	0.531	2.9	2.98
Biphenyl	5.0	1.43	1.2	0.162	2.9	1.25
Acenaphthylene	5.0	1.02	1.2	0.186	2.9	1.04
Acenaphthene	5.0	1.17	1.2	0.150	2.9	1.06
Dibenzofuran	5.0	1.36	1.2	0.201	2.9	2.29
Fluorene	5.0	1.02	1.2	0.138	2.9	3.02
C1-Fluorenes	5.0	1.02	1.2	0.138	2.9	3.02
C2-Fluorenes	5.0	1.02	1.2	0.138	2.9	3.02
C3-Fluorenes	5.0	1.02	1.2	0.138	2.9	3.02
Anthracene	5.0	1.27	1.2	0.225	2.9	0.726
Phenanthrene	5.0	1.20	1.2	0.246	2.9	2.62
C1-Phenanthrenes/Anthracenes	5.0	1.20	1.2	0.246	2.9	2.62
C2-Phenanthrenes/Anthracenes	5.0	1.20	1.2	0.246	2.9	2.62
C3-Phenanthrenes/Anthracenes	5.0	1.20	1.2	0.246	2.9	2.62
C4-Phenanthrenes/Anthracenes	5.0	1.20	1.2	0.246	2.9	2.62
Retene	5.0	0.681	1.2	0.174	2.9	0.600
Dibenzothiophene	5.0	1.23	1.2	0.144	2.9	1.28
C1-Dibenzothiophene	5.0	1.23	1.2	0.144	2.9	1.28
C2-Dibenzothiophene	5.0	1.23	1.2	0.144	2.9	1.28
C3-Dibenzothiophene	5.0	1.23	1.2	0.144	2.9	1.28

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Parameter	Whole Water (ng/L)		Sediment (ng/g dry)		Tissues (ng/g dry)	
	RL	MDL[2]	RL	MDL	RL	MDL
C4-Dibenzothiophene	5.0	1.23	1.2	0.144	2.9	1.28
Fluoranthene	5.0	0.682	1.2	0.444	2.9	0.944
Pyrene	5.0	0.531	1.2	0.537	2.9	0.848
C1-Fluoranthenes/Pyrenes	5.0	0.531	1.2	0.537	2.9	0.848
C2-Fluoranthenes/Pyrenes	5.0	0.531	1.2	0.537	2.9	0.848
C3-Fluoranthenes/Pyrenes	5.0	0.531	1.2	0.537	2.9	0.848
C4-Fluoranthenes/Pyrenes	5.0	0.531	1.2	0.537	2.9	0.848
Benzo(a)anthracene	5.0	0.610	1.2	0.369	2.9	0.752
Chrysene	5.0	0.710	1.2	0.333	2.9	0.606
C1-Chrysenes	5.0	0.710	1.2	0.333	2.9	0.606
C2-Chrysenes	5.0	0.710	1.2	0.333	2.9	0.606
C3-Chrysenes	5.0	0.710	1.2	0.333	2.9	0.606
C4-Chrysenes	5.0	0.710	1.2	0.333	2.9	0.606
Benzo(b)fluoranthene	5.0	0.883	1.2	0.390	2.9	0.723
Benzo(k)fluoranthene	5.0	0.839	1.2	0.201	2.9	0.663
Benzo(e)pyrene	5.0	0.798	1.2	0.303	2.9	0.642
Benzo(a)pyrene	5.0	1.14	1.2	0.411	2.9	0.468
Perylene	5.0	2.48	1.2	0.354	2.9	0.612
Indeno(1,2,3-cd)pyrene	5.0	0.808	1.2	0.462	2.9	0.543
Dibenz(a,h)anthracene	5.0	1.09	1.2	0.285	2.9	0.279
Benzo(g,h,i)perylene	5.0	0.990	1.2	0.450	2.9	0.417

Notes:

[1] Actual detection limits (e.g., LOD, MDL) may be higher or lower, depending on the specific analytical laboratory under contract at the time of project implementation.

[2] Laboratory MDLs are verified and updated periodically according to the organization's quality system. MDLs current at the time of analysis may vary from those defined in this QAPP. The actual laboratory MDLs and RLs will be reported with the laboratory data. Water MDL/RL based on a 1L sample size with a dilution factor of 1 and a PIV = 500uL. Sediment MDL based on a 20g sample size (wet weight; 16.63 g dry weight) with a dilution factor of 2 and PIV = 1000uL. Tissue MDL based on 20 g sample size (wet weight; 3.58 g dry weight) with a dilution factor of 2.05L and PIV = 500uL.

[3] Bolded compounds are the 16 priority PAH pollutants.

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Table 3-12 List of Saturated Hydrocarbons Target Analytes with Reporting Limits and Method Detection Limits[1]

Parameter	Whole Water (ng/L)		Sediment and Drilling Mud/Cuttings (ng/g dry)		Tissues (ng/g dry)	
	RL	MDL[2]	RL	MDL	RL	MDL
n-Nonane	500	43.1	120	7.46	305	28.7
n-Decane	500	54.2	120	5.70	305	90.2
n-Undecane	500	31.4	120	5.93	305	35.8
n-Dodecane	500	31.9	120	6.42	305	30.4
n-Tridecane	500	24.1	120	7.57	305	21.5
Isoprenoid RRT 1380	NA[3]	NA	NA	NA	305	NA
n-Tetradecane	500	22.7	120	5.66	305	29.6
Isoprenoid RRT 1470	NA	NA	NA	NA	305	NA
n-Pentadecane	500	26.5	120	5.49	305	56.3
n-Hexadecane	500	36.3	120	12.9	305	30.1
Norpristane (1650)	NA	NA	NA	NA	305	NA
n-Heptadecane	500	35.5	120	5.71	305	48.0
Pristane	500	31.3	120	25.0	305	95.3
n-Octadecane	500	50.5	120	8.06	305	23.9
Phytane	500	33.2	120	9.23	305	24.9
n-Nonadecane	500	38.5	120	4.91	305	26.9
n-Eicosane	500	46.8	120	10.1	305	30.6
n-Heneicosane	500	54.1	120	3.07	305	29.3
n-Docosane	500	58.9	120	17.6	305	36.3
n-Tricosane	500	43.5	120	6.97	305	25.8
n-Tetracosane	500	63.6	120	17.4	305	25.1
n-Pentacosane	500	69.8	120	15.4	305	16.2
n-Hexacosane	500	72.6	120	10.2	305	15.4

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Parameter	Whole Water (ng/L)		Sediment and Drilling Mud/Cuttings (ng/g dry)		Tissues (ng/g dry)	
	RL	MDL[2]	RL	MDL	RL	MDL
n-Heptacosane	500	69.5	120	8.97	305	25.9
n-Octacosane	500	78.9	120	9.62	305	19.7
n-Nonacosane	500	58.3	120	6.26	305	23.0
n-Triacontane	500	57.7	120	6.85	305	21.1
n-Hentriacontane	500	55.6	120	4.05	305	40.0
n-Dotriacontane	500	52.7	120	5.74	305	25.7
n-Tritriacontane	500	58.1	120	2.87	305	23.9
n-Tetratriacontane	500	47.0	120	4.99	305	30.6
n-Pentatriacontane	500	50.0	120	5.19	305	26.8
n-Hexatriacontane	500	45.4	120	4.44	305	31.1
n-Heptatriacontane	500	48.1	120	9.92	305	40.0
n-Octatriacontane	500	43.2	120	5.65	305	44.3
n-Nonatriacontane	500	49.9	120	9.26	305	52.6
n-Tetracontane	500	52.4	120	10.0	305	60.6
SHC Total (C9-C40)	NA	NA	NA	NA	NA	NA
TPH Total (C9-C40)	NA	9200	NA	694	NA	267,000

Notes:

[1] Actual detection limits (e.g., LOD, MDL) may be higher or lower, depending on the specific analytical laboratory under contract at the time of project implementation.

[2] Laboratory MDLs are verified and updated periodically according to the organization's quality system. MDLs current at the time of analysis may vary from those defined in this QAPP. The actual laboratory MDLs and RLs will be reported with the laboratory data. Water MDL based on 1L sample with a dilution factor of 1 and pre-injection volume (PIV) = 500uL. Sediment MDL based on a 20g sample size (wet weight; 16.63 g dry weight) with a dilution factor of 2 and PIV = 1000uL. Tissue RL is estimated based on 20 g sample size (wet weight; 3.36 g dry weight) with a dilution factor of 2.05L and PIV = 500uL.

[3] NA = standards not available for this compound; the RL and MDL of the compound which elutes prior to the isoprenoid compound is applied.

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Water samples will be analyzed for VOCs using gas chromatography by purge-and-trap techniques (Table 3-13). Samples are spiked with SIS and RIS and an inert gas is bubbled through the water samples. The VOCs are purged from the aqueous phase to the gas phase, which is swept through a sorbent trap where the VOCs are trapped, heated and back flushed with inert gas to desorb the components onto a non-polar fused silica capillary chromatographic column, separated via capillary gas chromatography, and identified and quantified using electron ionization mass spectrometry in the Full Scan mode. Target compounds will be quantified by using the method of internal standards using the SIS compounds resulting in surrogate recovery-corrected data generated to represent the native sample concentrations.

Table 3-13 List of VOC Target Analytes with Reporting Limits and MDLs[1]

Parameter	Whole Water (ng/L)		Drilling Muds and Cuttings (ng/g)	
	RL	MDL[2]	RL	MDL
1,4-Dichlorobenzene[3]	5.0	0.984	1.2	0.0737
1,3-Dichlorobenzene[3]	5.0	0.984	1.2	0.0737
1,2-Dichlorobenzene[3]	5.0	0.984	1.2	0.0737
Benzene	2950	564	11.8	1.21
Chlorobenzene[3]	TBD[2,3]	TBD	TBD	TBD
Ethylbenzene	2860	506	11.4	1.62
Toluene	1930	348	7.69	1.08
m-xylene	938	227	3.70	0.603
p-xylene	1990	311	7.94	1.20
o-xylene	957	180	3.82	0.528
Total Aromatic Hydrocarbons (TAH)	NA	NA	NA	NA

Notes:

[1] Actual detection limits (e.g., LOD, MDL) may be higher or lower, depending on the specific analytical laboratory under contract at the time of project implementation.
[2] Laboratory MDLs are verified and updated periodically according to the organization's quality system. MDLs current at the time of analysis may vary from those defined in this QAPP. The actual laboratory MDLs and RLs will be reported with the laboratory data. Water MDL based on a 0.02 L sample size and a dilution factor of 1. Sediment MDL based on a 5g sample size and a dilution factor of 1.
[3] These parameters may be analyzed by GC/MS purge and trap (SOP 5-245) or GC/MS SIM (SOP 5-157). MDLs are for GC/MS SIM using 20 g wet wt sediment (16 g dry weight) and 1L of water.
[3] To be determined (TBD). Laboratory MDLs will be developed and reported with the study data.

3.5.3.2 Hydrocarbons in Sediment Samples (Phases III and IV)

Samples of sediments, drilling muds, and cuttings will be extracted for PAH (Table 3-11), SHC and TPH (Table 3-12), and petroleum biomarkers St/Tr (Table 3-14), following laboratory SOPs. Muds and cuttings will also be analyzed for VOCs. Briefly, approximately 30 g of wet sediment will be fortified with SIS compounds and serially extracted with DCM. Less material may be used for the analyses of used drilling mud and cuttings because they may contain higher hydrocarbon concentrations than native sediment.

The sample extract will be dried and concentrated over anhydrous sodium sulfate. The extract will be purified with activated copper to remove any sulfur that is present and then will be purified further with an alumina clean-up column. The extract then will be purified further using silica gel column fraction to

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isolate the TPH/SHC fraction and petroleum biomarker fraction (F1) from the aromatic hydrocarbon fraction (F2). The resulting extracts will be concentrated and spiked with IS compounds. The TPH/SHC and petroleum biomarker F1 extracts will be submitted for TPH and SHC analysis by modified EPA Method 8015 using GC/FID and for petroleum biomarkers analysis by modified EPA Method 8270 using GC/MS-SIM. The F2 extract will be submitted for PAH analysis also using the modified EPA Method 8270 by GC/MS-SIM analysis. Target compounds will be quantified by using the method of internal standards using the SIS compounds resulting in surrogate recovery-corrected data generated to represent the native sample concentrations. The laboratory also will determine the sediment grain size and the total organic carbon (TOC) content of the sediments. Sediments will be processed by the sieve and pipette methods to determine sediment grain size with data for the following four fractions: gravel, sand, silt, and clay. For organic matter, the TOC content will be measured at 900ºC with a total-carbon analyzer.

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Table 3-14 List of Petroleum Biomarker (St/Tr) Target Analytes with Reporting Limits and Method Detection Limits[1]

Compound	Synonym	Sediment (ng/g dry)		Tissue (ng/g dry)	
		RL	MDL[2]	RL	MDL
C_{23} Diterpane	T4-C23Diterpane	1.8	0.318	7.6	1.39
13β,17α-diacholestane(20S)	S4-Diacholestane	0.60	0.114	2.5	0.402
13β,17α-diacholestane(20R)	S5-Diacholestane	0.60	0.114	2.5	0.402
C_{29} Tricyclictriterpane(20S)	T9-C29Tricyclictriterpane	1.8	0.318	7.6	1.39
C_{29} Tricyclictriterpane(20R)	T10-C29Tricyclictriterpane	1.8	0.318	7.6	1.39
5α,14α,17α-cholestane(20R)	Cholestane	0.60	0.114	2.5	0.402
18α(H)-22,29,30-trisnorhopane(TS)	T11-Trisnorhopane(TS)	1.8	0.318	7.6	1.39
17α(H)-22,29,30-trisnorhopane(TM)	T12-Trisnorhopane(TM)	1.8	0.318	7.6	1.39
5α,14α,17α,24-methylcholestane(20R)	S24-Methylcholestane	0.60	0.114	2.5	0.402
5α,14α,17α,24-ethylcholestane(20S)	S27-Ethylcholestane(S25)	0.60	0.114	2.5	0.402
5α,14α,17α,24-ethylcholestane(20R)	S28-Ethylcholestane(S28)	0.60	0.114	2.5	0.402
17α(H),21β(H)-30-norhopane	T15-Norhopane	1.8	0.318	7.6	1.39
18α(H)-oleanane	T18-Oleanane	1.8	0.318	7.6	1.39
17α(H),21β(H)-hopane	T19-Hopane	1.8	0.318	7.6	1.39
22S-17α(H),21β(H)-30-homohopane	T21-Homohopane	1.8	0.318	7.6	1.39
22R-17α(H),21β(H)-30-homohopane	T22-Homohopane	1.8	0.318	7.6	1.39
17β(H),21β(H)-hopane	17β(H),21β(H)-hopane	1.8	0.318	7.6	1.39

Notes:

[1] Actual detection limits (e.g., LOD, MDL) may be higher or lower, depending on the specific analytical laboratory under contract at the time of project implementation.

[2] Laboratory MDLs are verified and updated periodically according to the organization's quality system. MDLs current at the time of analysis may vary from those defined in this QAPP. The actual laboratory MDLs and RLs will be reported with the laboratory data. Sediment MDL based on a 20g sample size (wet weight; 16.63 g dry weight) with a dilution factor of 1 and PIV = 1000uL. Tissue RL was estimated based on a 20g sample size (wet weight; 4.03 g dry weight) with a dilution factor of 2.05L and PIV = 1000uL. In the event that additional petroleum biomarkers are included in analysis, RLs and MDLs can be provided by the laboratory upon request.

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3.5.3.3 Hydrocarbons in Tissue Samples (Phases III and IV)

Samples of biological tissues will be extracted for PAH (Table 3-11), SHC and TPH (Table 3-12), and petroleum biomarkers St/Tr (Table 3-14) following laboratory SOPs. About 20 g of wet tissue will be fortified with SIS compounds and serially extracted with DCM. The tissue will be extracted by maceration using a TissueMizer® or equivalent, with stainless-steel probes. The sample extract will be dried and concentrated over anhydrous sodium sulfate. The extract will be purified with an alumina clean-up column. The extract then will be purified further with silica-gel column fraction to isolate the TPH/SHC fraction and petroleum biomarker fraction (F1) from the aromatic hydrocarbon fraction (F2). The resulting extracts will be concentrated and spiked with IS compounds. The TPH/SHC and petroleum biomarker F1 extracts will be submitted for TPH and SHC analyses by modified EPA Method 8015 using GC/FID and for analysis of petroleum biomarkers by modified EPA Method 8270 using GC/MS-SIM. The F2 extract will be submitted for PAH analysis using the modified EPA Method 8270 by GC/MS-SIM analysis. Target compounds will be quantified by using the method of internal standards using the SIS compounds resulting in surrogate recovery-corrected data generated to represent the native sample concentrations. The laboratory also will determine the lipid content of the tissue, based on the total extractable material (TEM).

3.5.4 Macrofaunal Analysis

Sediment samples will be processed shipboard by sieving according to methods outlined in Section 3.5.1.8, and taxonomic analysis will be conducted on infaunal invertebrates to determine community composition. In the laboratory, receipt of benthic samples will be logged and processing steps recorded in a notebook to track each sample. Samples will be kept in formalin for a minimum of 1 month, and preferably no longer than 4 months, to prevent decalcification of small bivalve shells. After 1–3 months, the formalin in benthic samples will be decanted and the samples rinsed and then preserved in isopropyl alcohol (or 70% ethanol). Decanted formalin and rinsing water will be poured through a 0.5-mm mesh screen or smaller to capture any specimens that otherwise would be lost. Biological tissues in the 1.0-mm mesh sieve samples will be sorted from the sediment remains into major taxonomic units, and the invertebrate animals will be identified to the lowest practical taxonomic category. A small sample “tag” will be created on waterproof paper for each group of specimens of similar taxonomic determination to follow that group through identification and weighing. Samples will be weighed for wet-weight biomass and stored in alcohol in a glass container along with the specimen tags. Data will be entered into a computer with a database program. After analysis, samples should be moved to glass containers for long-term storage and archival. Lids will be sealed with plastic liners, electrical tape or parafilm to reduce the rate of evaporation of fluids.

Resulting metrics include taxonomic identification, abundance (individuals m^{-2}), and biomass (g m^{-2}). The data on benthic density and biomass resulting from taxonomic analysis will be used for statistical analysis. A small subset of samples employed for community analysis should be reprocessed by a different technician or multiple technicians, when appropriate. Where questions arise, specimens will be compared with the authoritative voucher sets or sent to an appropriate taxonomic expert. Quality-control methods for benthic taxonomic analysis will follow guidelines outlined in SOP SAM043.01 *Benthic Sample Collection and Processing for Shell Baseline Environmental Sampling Program – Chukchi Sea*.

3.5.5 Sediment Profile Imaging Analysis

Table 3-15 below identifies the range of parameters that may be evaluated from the sediment photos. Further descriptions of the parameter groups are provided below.

Table 3-15 Sediment Profile Imaging Parameters

Parameter	Units	Method	Description
Sediment grain size	Modal phi φ interval	Visual analysis (V)	An estimate of sediment types present. Determined from comparison of images of known grain size.
Prism penetration	cm	Computer analysis (CA)	A geotechnical estimate of sediment compaction. Average of maximal and minimal distances from sediment surface to bottom of prism window.
Sediment surface relief	cm	CA	An estimate of small-scale bed roughness. Maximal depth of penetration minus minimal depth.
Apparent Redox Potential Discontinuity depth (from color change in sediment)	cm	CA	Estimate of depth to which sediments appear to be oxidized. Area of aerobic sediment divided by width of digitized image
Thickness of sediment layers	cm	CA	Measure thickness above original sediment surface
Methane/Nitrogen Gas Voids	number	V	Count
Epifaunal Occurrence	number	V	Count, identify
Tube Density	number/cm^2	V	Count
Tube Type			
Burrow Structures	—	V	Identify
Pelletal Layer	cm	V	Measure thickness, area
Bacterial Mats	—	V	Determine presence and color
Infaunal occurrence	number	V	Count, identify
Feeding Voids	number	V	Count, measure thickness, area
Apparent Successional Stage	—	V,CA	Estimated based on all of the above parameters

Sediment Grain Size—This parameter is a textural feature of the sediment used to describe the type of sediment present. Based on grain-size distribution, the nature of the physical forces acting on a habitat can be inferred. Grain size is determined by comparison of collected images with a set of standard images for which mean grain size has been determined in the laboratory.

Prism Penetration—This parameter provides an indication of sediment stiffness, with the profile camera prism acting as a penetrometer. The further the prism enters into the sediment, the softer the sediment is and typically indicates higher water content. Penetration is measured as the distance from the leading (deeper) edge of the prism to the sediment-water interface, up the length of the face plate. If the weight of the camera frame is not changed during image collection, the prism penetration provides a means for assessing the relative compaction between stations. In the unlikely event adjustments to weight and/or flotation are made, a minimum of three replicate drops with both system weight/flotation setups will be made to provide corrections for prism penetration calculations.

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Surface Relief—Surface relief is measured as the difference between the maximal and minimal penetration measurements recorded from the sea bottom in one image. Another way to describe this measurement is the distance between the 'peaks' and 'valleys' of the sea bottom within the image. This parameter provides an estimate of small-scale bed roughness, on the order of the width of the prism's face plate (15 cm).

Apparent Color Redox Potential Discontinuity (SPI RPD) Layer—This parameter is used as an estimator of benthic habitat quality. It is the depth to which sediments are oxidized. The term "apparent" is used in describing this parameter because no actual measurement is made of the redox potential. An assumption is made that, given the complexities of iron and sulfate reduction-oxidation chemistry, reddish-brown sediment color tones (Diaz and Schaffner 1988) are indications that the sediments are oxic or at least are not intensely reducing. This assumption is in accordance with the classical concept of RPD depth, which associates it with sediment color (Fenchel 1969, Lyle 1983). The depth of the apparent color RPD is defined as the area of all the pixels in the image discerned as being oxidized divided by the width of the digitized image.

Surface Features—A variety of physical and biological features can be seen at or on the sediment surface. These features can range from SAV, worm tubes, fecal pellets, epibenthic organisms, bacterial mats, algal mats, shells, mud clasts, and bed forms to feeding pits and mounds. Each of these features provides information on the type of habitat and its quality. Surface features are evaluated visually from each slide and compiled by type and frequency of occurrence.

Subsurface Features—Subsurface features include a wide variety of features (such as infaunal organisms, burrows, water-filled voids, gas voids, or sediment layering) and reveal information about physical and biological processes influencing the sea floor. Subsurface features also provide data about the physical–biological control occurring in a habitat.

Successional Stage—Sediment profile data can be used to estimate successional stage of the fauna in a habitat (Rhoads and Germano 1986). Successional stage is based on the theory that organism-sediment interactions follow a predictable sequence after a major seafloor disturbance (Rhoads and Germano 1982). Characteristics that are associated with pioneering or colonizing (Stage I) assemblages (Pearson and Rosenberg 1978), such as dense aggregations of small polychaete tubes at the surface and shallow apparent RPD layers, may be readily visible in sediment-profile images. Advanced or equilibrium (Stage III) assemblages also have characteristics that can be seen in profile images, such as deep apparent RPD layers and subsurface feeding voids. Stage II is intermediate between Stages I and III and has characteristics of both (Rhoads and Germano 1986).

ROV Images-Once the survey is complete, the photos and video data will be sorted, analyzed and archived by the science crew. The videos and photos are directly interpreted by benthic scientists and observations recorded on forms provided for this purpose. A data sheet is filled out for each video.

3.5.6 Laboratory Instrument/Equipment Calibration, Maintenance, and Operation

Laboratory instruments and equipment must be tested, maintained, and calibrated according to SOPs and the manufacturers' instructions prior to use. The manufacturer manuals and SOPs must be readily available at the bench so that trouble-shooting and routine repairs can be performed correctly. All routine

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maintenance and non-routine repairs are to be documented in a permanent location (e.g., electronically or in bound logbooks). The return to analytical control is demonstrated by successful calibration. Instruments and equipment that are out-of-calibration must be tagged or removed from the laboratory to prevent inadvertent use. Calibration requirements are defined in Table 3-16.

- Certified calibration standards will be used for instrument calibration. Where possible, standards will be traceable to National Institute of Standards and Technology (NIST).
- Stock solutions for spiking solutions, surrogate compounds, and other organic or inorganic compound mixes will be made from reagent-grade chemicals or as specified in the SOPs. All analytical stock solutions will be prepared using Class-A volumetric ware.
- Preparation of stock solutions must be documented including the parent material used (lot number and concentration), the amount used, the final solution volume, and the final solution concentration. Specific handling and documentation requirements for the use of standards will be defined in laboratory SOPs.
- All new calibration or spiking solutions must be analyzed against a previously accepted standard to verify that the concentrations are acceptable.

Prior to analysis, a calibration curve must be verified through the analysis of a check solution prepared from a source (or at least a lot) independent of that used for the initial calibration curve. The calibration check solutions must include all targeted analytes.

Table 3-16 Calibration Procedures for Laboratory Instruments

Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action (CA)
Gas Chromatography/ Mass Spectrometry (GC/MS)	Initial Calibration (ICAL) Standard	Prior to samples	≤25% relative standard deviation (RSD) for each analyte and average RSD for all analytes ≤ 15%, R≤0.995	Reanalyze, if failure repeats, take remedial action, if failure continues, a new ICAL is performed
	Independent Calibration Check (ICC)	After ICAL or if failure of continuing calibration verification (CCV)	PD must be ≤ 25% for each analyte and internal standard (IS) area has not changed by more than a factor of two from the area in level 3 ICAL	Evaluate and re-analyze, if this ICC fails, perform remedial action and reanalyze. If a second ICC fails, a new ICAL is performed or justify results.
	CCV	Beginning and end of 10 injections, or 24 hr period	≤25% from true check std. conc. for each analyte and average difference for all analytes ≤ 15%, and IS area has not changed by more than a factor of two from the area in level 3 ICAL	Re-injection of samples prior to a CCV may be warranted. Evaluate samples, sample extracts must be bracketed by an acceptable CCV
GC-Flame Ionization Detection (FID)	ICAL Standard	Prior to samples	≤25% relative standard deviation (RSD) for each analyte and average RSD for all analytes ≤ 20%, R≤0.995	Evaluate, re-analyze if necessary, discuss with PM
	ICC	After ICAL	RSD must be ≤ 25% for each analyte and ≤ 25% for the mean, the IS area must be within a factor of 2 from the Level 3 ICAL and mass discrimination must be ≥0.8	Reanalyze, if failure repeats, take remedial action, if failure continues, a new ICAL is performed
	CCV	After every 10 injections, or 24 hr	≤25% from true check standard conc. for each analyte and average difference for all analytes ≤ 20%	Reanalyze, if failure repeats, take remedial action, if failure continues, a new ICAL is performed. Samples must be bracketed by an acceptable CCV
Purge and Trap GC/MS	ICAL Standard	Prior to samples	≤30% RSD for each analyte and average RSD for all analytes ≤ 15%, R≤0.995	Recalibrate if >10% of target analytes exceed %RSD or R^2 criteria If ≤ 10% of target compounds exceed criteria, recalibration is not required provided %RSD is < 40% or R^2 >0.98. If failure repeats, take remedial action, if failure continues, a new ICAL is performed

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Instrument	Calibration Procedure	Frequency	Acceptance Criteria	Corrective Action (CA)
Purge and Trap GC/MS (continued)	ICC	After ICAL	PD must be ≤ 30% for most analytes (40-60% for oxygenates) and the IS area must be within a factor of 2 from the Level 3 ICAL	Recalibrate if >10% of target analytes exceed criteria. Reanalyze, perform remedial action as necessary, reanalyze if second ICC fails, a new ICAL is performed
	CCV	beginning and end of each 12 h period	≤30% PD for each analyte, average difference for all analytes ≤ 15% and the IS area must be within a factor of 2 from the Level 3 ICAL	Reanalyze if >10% of target analytes exceed criteria. If ≤ 10% of target compounds exceed criteria, affected sample re-analysis is not required if the % difference for exceeding analytes is < 40% (or 60% for oxygenates) Evaluate and re-analysis of samples analyzed after failed CCV, if necessary. Samples must be bracketed by an acceptable CCV
CVAAS	5 point curve	Before, during, and after analysis of 20 samples	R^2>0.995	Recalibrate
Total Hg cold vapor atomic fluorescence spectrophotometer (CVAFS)	5 point curve using two standards	Daily	RSD<15%	Recalibrate
Methylmercury CVAF	5 point curve using two standards	Daily	RSD<15%	Recalibrate
ICP-MS	3-5 point curve depending on metal; r≥0.999 for all metals	Recheck standards every 8-10 samples	Continuing calibration with %RSD <15%	Rerun samples
FAAS	3-5 point curve depending on metal; r≥0.999 for all metals	Recheck standards every 8-10 samples	Continuing calibration with %RSD <15%	Rerun samples
Total Carbon Analyzer	4 pt curve, r≥0.999	Recheck standards every 8-10 samples	Continuing calibration %RSD <15%	Rerun samples

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3.6 Quality Assurance/Quality Control

Personnel performing QA activities within each organization must be trained and independent of the technical work they are reviewing. QA staff will monitor the technical components of the project according to organizational SOPs to ensure the accuracy, integrity, and completeness of the data. Analytical staff members will be responsible for ensuring that sample tracking, sample preparation, and analytical instrument operation all meet the quality-control criteria detailed in the applicable analytical SOPs.

The project design incorporates SOPs to ensure consistency (Table 3-17) and QC procedures and checks in both the field and laboratory in order to assess data quality. The study design and QC samples are intended to assess the major components of total study error, which facilitates the final evaluation of whether environmental data are of sufficient quality to support the related decisions. The QC sample requirements are designed to provide information on measurement error that can be used to initiate corrective actions with the goal of limiting the total measurement error. Note: Possible analytical laboratories and associated SOPs are listed in Table 3-17. Laboratories may change prior to project implementation and may include other contract analytical laboratories (and subsequently slightly different SOP numbers and titles).

Table 3-17 Standard Operating Procedures

SOP Number	SOP Title
Field	
FWS SOP-01	Field Documentation
FWS SOP-02	Sample Custody
FWS SOP-03	Sample Packaging and Shipping
FWS SOP-04	Decontamination of Equipment – Sediments
FWS SOP-05	Preparation of Field Quality Control Samples
FWS SOP-06	Surface Sediment Sampling Using a Modified van Veen Grab Sampler
FWS SOP-07	Decontamination of Sampling Equipment
FWS SOP-08	Preparation of Field Quality Control Samples – Tissues
FWS SOP-12	Benthic tissue sampling for chemical analysis using a dredge/rake
FWS SOP-13	Amphipod sampling for chemical analysis using a modified minnow trap
SAM044.01	Sediment Profile Imaging and Plan View Photography Collection
SOP 5-275	At-Sea Collection of Hydrographic Data Using CTD and Rosette System
SOP 5-342	Collecting Sediment Cores with a Piston Push/Hammer Corer
SAM043.01	Benthic Sample Collection and Processing
FIT-0001	Field Collection of Seawater Samples for Dissolved Trace Metals
FIT-6001	Filter Preparation for Suspended Matter Collection and Trace Metal Analysis
FIT-6002	Collection and Digestion of Particulate Samples for Trace Metal Analysis

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SOP Number	SOP Title
Laboratory	
SOP 5-157	Identification and Quantification of Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry
SOP 5-190	Tissue Extraction for Trace Level Semi-Volatile Organic Contaminant Analysis
SOP 5-191	Size Exclusion high performance liquid chromatography (HPLC) Cleanup of Sample Extracts for Semi-Volatile Organic Pollutants
SOP 5-192	Soil/Sediment Extraction Using an Orbital Shaker Table Method for Trace Level Semi-Volatile Organic Contaminant Analysis
SOP 5-200	Water Extraction for Trace Level Semi-Volatile Organic Contaminant Analysis
SOP 5-202	Determination of Low Level Total Petroleum Hydrocarbons and Individual Hydrocarbon Concentrations in Environmental Samples
SOP 5-245	Preparation and Analysis of Volatile Hydrocarbons in Environmental Samples
SOP 5-328	Removal of Sulfur from Environmental Sample Extracts
SOP 5-330	Silica Gel Fractionation of Environmental Extracts for the Separation of Saturated Hydrocarbons and Aromatic Compounds
SOP 5-329	Alumina Clean-up of Environmental Sample Extracts
SOP 6-010	Sample Receipt, Custody, and Handling
FIT-2010-GS	Grain size by sieve and pipette
FIT-0004 Series	Determination of (Metals) in Seawater Following Preconcentration by Reductive Precipitation
FIT-0005 Series	Determination of (Metals) in Seawater Following Preconcentration using SeaFast column
FIT-1012 Series	Determination of (Metals) in Tissues ICP-MS
FIT-1013 Series	Determination of (Metals) in Tissues by FAAS
FIT-2010 Series	Determination of (Metals) in Sediments by FAAS
FIT-2012 Series	Determination of (Metasl) in Sediments by ICP-MS
FIT 2015	Determination of Total Organic Carbon
FIT-6003	Determination of Particulate (Metals) by FAAS
FIT-6005	Determination of Particulate (Metals) by ICP-MS
MSL-I-013	Total Mercury in Aqueous Samples by Cold Vapor Atomic Fluorescence (CVAF)
MSL-I-014	Methylmercury in Aqueous Samples by Cold Vapor Atomic Fluorescence (CVAF)
MSL-I-015	Methylmercury in Tissues and Sediments by Cold Vapor Atomic Fluorescence (CVAF)
MSL-I-034	Determination of Mercury in Tissues and Sediments by Direct Thermal Decomposition and Cold Vapor Atomic Absorption Spectrometry (CVAAS)
MSL-I-034-02	Determination of Mercury in Tissues and Sediments by Direct Thermal Decomposition and Cold Vapor Atomic Absorption Spectrometry (CVAAS)
SAM043.01	Benthic Sample Collection and Processing

3.6.1 Field Quality Control

Field QC samples will be collected in the same type of sample containers and in all other ways handled in the same manner as other field samples. The field QC samples will be assigned unique sample numbers

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and will be submitted to the analytical laboratory as routine samples. If abnormalities are detected in field QC samples, the data associated with the QC samples will be assessed to determine if project data are affected. FWS SOP-05 *Preparation of Field Quality Control Samples* and FWS SOP-08 *Preparation of Field Quality Control Samples – Tissue* describe specific procedures for preparing field QC samples.

The following field QC samples will be collected:

- Trip blanks will be prepared and submitted for analysis at the frequency of one per shipping container of VOC samples.
- Temperature blanks will accompany each cooler that contains samples with a temperature preservative requirement. The temperature blank will be created either in the field or laboratory by filling polyethylene bottles with deionized water or sediment, depending on the matrix being shipped.
- Field duplicates will be collected as separate co-located sample and will be analyzed for the same chemical and physical parameters as the other samples from its location. At a minimum, field duplicates are collected at 5% each of the sediment, water, and tissue samples.

Field QC samples that will be collected specific to the EMP are described in the following sections.

3.6.1.1 Equipment (Rinsate) Blank (EB)

An equipment blank is a sample of contaminant-free medium (typically reagent-grade water) that has been passed through or over the sampling equipment used to collect field samples. An equipment blank is collected in the same type of sample containers and in all other ways is handled in the same manner as other field samples. The equipment blank must be collected during the sampling event, after collection of at least one field sample, after normal equipment decontamination procedures, and prior to collection of the next field sample. Sampling devices for sediment include the grab, stainless steel spoons or spatulas, bowls, and flexible tubing. EB will be collected at a frequency of 5% of the total number of field samples collected for sediment and water.

3.6.1.2 Field Blanks

Field blanks will be collected in the field, depending on the type of associated analysis. For example, a sample of analyte free water poured into the container in the field, preserved and shipped to the laboratory with field samples serves to assess contamination from field conditions during sampling. Additionally, filter blanks will be collected for the dissolved metals to estimate any contamination associated with the field filtering process. Field blanks are prepared at a frequency of 5% of the total number of field samples collected for sediment and water.

3.6.1.3 Laboratory Duplicates (Laboratory Splits)

Laboratory duplicates (also called laboratory splits) are used to assess the precision of the analytical method and laboratory handling. For the laboratory duplicate analysis, one sample will be split by the analytical laboratory into two portions and each analyzed. When collecting samples to be analyzed for laboratory duplicates, typically double the normal sample volume is required. This requires filling a larger size sample bottle, or filling two normal size sample bottles and labeling one with the site name and the second with the site name plus “laboratory duplicate”. Laboratory duplicate samples are collected,

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handled, and delivered to the analytical laboratory in the same manner as environmental samples. In some cases, laboratory duplicates may be replaced by matrix spike and matrix spike duplicate samples, which also are indicative of precision of the analytical method and laboratory handling.

3.6.1.4 Benthic Samples

For benthic samples, every effort will be made to use only grabs that have demonstrated no wash-out of the sediment surface. The quantity of sediment retained in the van Veen grab buckets should range from full to minimum penetration depth regardless of the material type and/or coarseness. Samples with insufficient penetration and low-volume of sediment recovery are rejected for community comparison samples. Samples will be carefully dropped into buckets and transported and poured into the sieves without significant spillage. A substantial spillage will require a new sample. Samples will be rinsed over a 1.0-mm mesh with a water pressure strong enough to remove sediments, but weak enough to minimize damage to the animals (nested sampling with a stacked sieve with 0.5-mm mesh may also be used). Biological and sediment residues will be carefully removed from each sieve so that all visible animals, sediment particles, and fragments will be removed. Samples then will be placed into Whirl-Pak® bags or jars labeled with the date of collection, station name, replicate number, sieve mesh size, and initials of personnel packaging the samples. Pertinent information also will be recorded on sample collection sheets listing the collection date, station name, water depth, and gross sediment characteristics. This information will also be included on a waterproof sample tag that is placed inside the specimen jar or container. The pertinent information then will be entered by the field personnel into the shipboard database for managing workflow and data collections and will be checked by the field lead daily for accuracy.

3.6.1.5 Accuracy and Precision of the ADCP and CTD Sensor Arrays

Table 3-18 defines typical accuracy and precision of ADCP instrument sensors. Accuracy and precision are determined based on the pre-season calibration check and post-season calibration check. Table 3-19 defines typical accuracy and precision of the CTD instrument sensors. The accuracy and precision of the equipment deployed during the environmental monitoring will be similar to these values and will be documented and reported with the data.

Table 3-18 Typical Accuracy and Precision of Instrument Sensors (ADCP and OBS)

Sensor	Model (or equivalent)	Measurement	Units	Range	Accuracy	Precision
ADCP	Teledyne RDI Workhorse Sentinel 300khz (or equivalent)	Current velocity	cm/s	0 to 500	1	0.1
		Depth	M	0 to 40	2%	1 cm
		Waves	cm, sec	> 0 cm > 3.3 sec	1%, 1%	1cm, 0.5 sec
OBS	Seapoint Sensors Turbidity Meter (or equivalent)	Turbidity	NTU	0 to 25	Calibration Dependent	2%

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Table 3-19 Typical Accuracy and Precision of Instrument Sensors (CTD)

Sensor	Model/Method (or equivalent)	Units	Range	Accuracy	Resolution
Pressure	Sea-Bird SBE 19*plus* V2 (or equivalent)	Decibars	0 to 1000	0.1% of range	0.002% of range
Temperature	Sea-Bird SBE 19*plus* V2 (or equivalent)	ºC	-5 to +35	0.005	0.0001
Conductivity	Sea-Bird SBE 19*plus* V2(or equivalent)	mS/cm	0 to 9000	0.5	0.05
Transmissometer (25-cm)	WET Labs C-Star 25cm (or equivalent)	m^{-1}	0 to 40	0.20	0.01
Turbidity	Seapoint Sensors Turbidity Meter (or equivalent)	NTU	0 to 25	Calibration Dependent	0.01
			0 to 125	5%	2%
			0 to 500	5%	2%
ADCP	Teledyne RDI Workhorse Sentinel 300khz (or equivalent)	Current velocity (cm/s)	0 to ± 2000	± 0.5% of water velocity relative to ADCP ± 0.5 cm/s	0.1
		Echo Intensity Profile (decibels)	0 to 80	1%	±1.5dB
		Depth(m)	0 to 165	2%	0.5m (bin size)

3.6.2 Laboratory Quality Control

Quality control is an integral part of the laboratory activities. It establishes methods for maximizing the quality of operations and analyses, provides analysts with metrics about method performance, and aids project managers in identifying and correcting systematic and random problems that can plague the laboratory operations.

Laboratory samples will be processed and analyzed in analytical batches or sample delivery groups (SDGs) of ≤20 field samples plus laboratory QC. A suite of QC samples that monitors the accuracy and precision of the methods will be incorporated into each batch as defined below. In addition to these QC samples, surrogate internal standards will be spiked into each sample analyzed for organic compounds. The acceptance criteria and corrective action for each QA sample is defined in Table 3-20.

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Table 3-19 Measurement Quality Criteria[1]

QC Sample Type	Measurement Quality Objective	Corrective Action
Field Duplicate (DU)	No criteria defined. Results will be used to assess sample heterogeneity or inconsistent sample handling and analysis procedures	
VOC (water/drill mud/ cuttings)		
Method Blank (MB)	Target analyte concentration in MB <5x the MDL. Data are acceptable if field sample concentration >5x MB; data <5x the MB will be flagged regardless of MB concentration	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e..g., "B" data qualifier, unless "J" flagged.
Field Trip Blank (TB)	Target analyte concentration in TB <5x the MDL.	Review with PI. If data fail MQO, report data with qualifiers; e.g., "B" data qualifier.
Laboratory Control Sample (LCS)	Spiked target analytes must be recovered at 50-130%	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Matrix Spike (MS)	Spiked target analytes must be recovered at 70-130%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Compare with LCS results. If the MS results are outside the LCS, review with the PI to determine if the difference is attributed to matrix effect or analytical error. Review all sample prep records, re-analyze as directed by the PI. If data fail MQO, report data with qualifiers; e.g., "MI" data flag.
Hydrocarbons (PAH/SHC/TPH/Biomarker)[2]		
Method Blank (MB)	Target analyte concentration in MB <5x the MDL, or MB result is N-qualified. Data are acceptable if field sample concentration >5x MB; however, field sample data <5x the MB will be B-qualified regardless of MB concentration.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e..g., "B" data qualifier, unless "J" flagged.
Laboratory Control Sample (LCS)	Target analyte recoveries:70-130%; 50-130% for nonane.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Matrix Spike (MS)	Target analyte recoveries:70-130%; 50-130% for nonane. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Compare with LCS. If the MS results are outside the LCS, review with the PI to determine if difference is due to matrix effects or analytical. Review sample prep records, re-analyze as directed by the PI. If data fail MQO, report data with qualifiers; e.g., "MI" data flag.
Matrix Spike Duplicates (MSD)	RPD ≤ 30%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "MI" data flag.
North Slope Crude (NSC)	RPD ≤ 30%. Concentration must be >5x the MDL for MQO to apply.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Standard Reference Material (SRM)	PD ≤ 30% from target concentration and the 95% confidence level. Analyte concentration must be certified and >5x the MDL for MQO to apply.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Surrogate Internal Standard (SIS)	Recoveries: 40-120%.	Review with PI, possibly re-analyze and/or re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.

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QC Sample Type	Measurement Quality Objective	Corrective Action
Metals (Except Mercury and Methyl Mercury)		
MB	<5x the MDL, or field sample concentration >10x MB.	Review with PI, possibly reanalyze. If all samples are >10x the MB, no corrective action is required. If samples are <10x the MB, the batch is re-prepped and re-analyzed. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Filter Blank (for dissolved metals) (FB)	<5x the MDL, or field sample concentration >10x MB.	Review with PI. If all samples are >10x the MB, no corrective action is required. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MS	Target recoveries: 80-120%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly reanalyze. Failure of multiple MQOs requires re-preparation and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MSD	RPD ≤ 20%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
SRM	Target recovery: 80-120% of certified value.	Review with PI, possibly reanalyze. Failure of multiple MQOs requires re-preparation and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Mercury		
MB	<5x the MDL, or field sample concentration >10x MB.	Review with PI, possibly reanalyze. If all samples are >10x the MB, no corrective action is required. If samples are <10x the MB, the batch is re-prepped and re-analyzed. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
OPR (mercury analysis only; one at start and one at end of batch; similar to LCS)	Target recoveries: 70-130% of (sediment and tissue) or 77-123% (water).	Review with PI, possibly re-prepare and reanalyze with all samples in associated batch for failed analytes. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MS	Target recoveries: 80-120% (sediment and tissue) or 71-125% (water). Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly reanalyze. Failure of multiple MQOs requires re-preparation and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MSD	RPD ≤ 20% (sediment and tissue) or ≤ 24% (water). Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
SRM	Target recovery: 80-120% of certified value (sediment and tissue) or 77-123% of certified value (water).	Review with PI, possibly reanalyze. Failure of multiple MQOs requires re-preparation and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Sensitivity	Low-level Check Sample analyzed at the MRL Target recovery: 80-120%. Concentration must be >5x the MDL for MQO to apply.	Review with PI, possibly re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.

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QC Sample Type	Measurement Quality Objective	Corrective Action
Methylmercury		
MB	<5x the MDL, or field sample concentration >10x MB.	Review with PI, possibly reanalyze. If confirmed and all samples are > 10x the blank, no corrective action is required. If samples are <10x the blank, the batch must be re-prepped and re-analyzed. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
OPR (one at start and one at end of batch; similar to LCS)	Target recovery: 67–133%.	Review with PI, possibly re-prepare and reanalyze with all samples in associated batch for failed analytes. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MS	Target recovery: 65-135%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple MQOs requires re-preparation and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
MSD	RPD ≤ 35%. Spike concentration must be >5x unspiked field sample concentration for MQO to apply.	Review with PI, possibly re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
SRM	Target recovery: 67-133% of certified value.	Review with PI, possibly reanalyze. Failure of multiple MQOs/OPRs requires redigestion and reanalysis of batch. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
Sensitivity	Low-level Check Sample analyzed at the MRL Target recovery: 80-120%. Concentration must be >5x the MDL for MQO to apply.	Review with PI, possibly re-extract and reanalyze. If data fail MQO, report data with qualifiers; e.g., "N" data flag.
TOC (Sediment)		
LCS	80 – 120% of certified value	Correct Problem, re-prepare and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, call PI.
QADU	RPD <10% at 10x detection limit	Review with PI, re-extract and reanalyze. If data continues to fail, report data with qualifiers.
SRM	±15% of certified value	Reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple CRMs requires redigestion and reanalysis of batch.
Completeness	>90%	Report results and assess impact
POC (Water Particulates)		
MB	All analytes in the method blank must be less than ½ the RL	Re-prepare and reanalyze the method blank and all samples processed with the contaminated blank. If problem persists, call PI.
LCS	80 – 120% of certified value	Correct Problem, re-prepare and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, call PI.
LCSD	RPB<10%	Review with PI, re-extract and reanalyze. If data continues to fail, report data with qualifiers.

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QC Sample Type	Measurement Quality Objective	Corrective Action
SRM	±15% of certified number	Reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple CRMs requires redigestion and reanalysis of batch.
Completeness	>90%	Report results and assess impact.
TSS (Water)		
MB	No target analyte concentrations ≥ RL or ≥ 10% of the measured values of the samples (whichever is larger)	Re-prepare and reanalyze the method blank and all samples processed with the contaminated blank. If problem persists, call PI.
LCS SRM	Analyte must have recoveries of 90-100% or be within the manufacturer's control limits if they are great than the 90-100% range	Correct problem, re-prepare and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, call PI.
LCSD	RPD ≤ 20%	Review with PI. If data continues to fail, report data with qualifiers.
Completeness	≥90%	Review with PI. If data continues to fail, report data with qualifiers.
Grain Size (Sediment)		
Laboratory Duplicate	RDP<20% of any individual values for gravel, sand, silt, or clay	Correct problem by re-homogenizing sediment to provide a more representative sample and repeat analysis.
Completeness	>90%	Assess impact and take corrective action.
Sediment Profile Imaging		
Independent Check	Measurements ±1.0 cm and Counts ± 2 counts Pixel density (adequate focus, exposure, and detail)	Reanalyze all images for parameters out of QC range.
Benthic Organisms (Sediment)		
Laboratory report	>90% for Overall precision, analytical precision, analytical Accuracy/Bias	Correct problem, resort, reweigh all samples in batch.
Taxonomy	100% of junior taxonomist samples verified. Questionable organisms of senior taxonomists verified	Discuss identifications with taxonomists.

Notes:

[1] Table represents criteria for all matrices unless otherwise noted.
[2] Hydrocarbon MQOs are based on the use of surrogate recovery corrected data.

The following laboratory QC samples will be analyzed:

- Method blank for each preparatory and analytical batch.
- LCS (prepared using contaminant-free matrix-specific sample, e.g., Ottawa sand or sodium sulfate [sediment] and clean Tilapia [tissue]).
- MS/MSD samples, prepared from the same parent sample and spiked with the analytes of interest at approximately 10X the MDL.
- A laboratory duplicate (QADU) is a second aliquot of a field sample processed and analyzed to monitor precision. It may be a second matrix-spike sample. As previously described, MS/MSD data may replace the laboratory duplicate sample analysis.

Additional laboratory QC specific to the EMP are described in the following sections.

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3.6.3 Standard Reference Material

An SRM is characterized by a metrologically valid procedure for one or more specified properties, accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability. A SRM is prepared with each processing batch for the appropriate sample matrix and analytical parameters (SRMs are not available for all parameters and sample types) to assess accuracy of the analytical procedures where applicable to a given matrix and analyte.

3.6.3.1 North Slope Crude Reference Oil

A NSC Reference Oil sample is used to evaluate the instrumental accuracy and also provide petroleum pattern information in the petroleum hydrocarbon analyses, aiding in the qualitative identification of target analytes. The NSC is analyzed for organics only.

The MQO for each QC sample in this project are presented in Table 3-20. Analytical results that do not meet the MQOs will be submitted to and/or reviewed with the PI for assessment of the potential impact of the results. Affected samples may be reanalyzed at the PI's discretion. QC sample data that are accepted outside the MQOs qualified, and the rationale for accepting the analysis will be documented.

3.6.3.2 Samples for Biological Community Analysis

A small subset of samples employed for community analysis will be reprocessed by a different technician or multiple technicians, when appropriate. Where questions arise, specimens will be compared with the authoritative voucher sets, sent to an appropriate taxonomic expert. Quality-control methods for benthic taxonomic analysis will follow guidelines adapted from the US EPA's Environmental Monitoring and Assessment Program (www.epa.gov/emap/html/pubs/docs/groupdocs/estuary/field/labman.html).

3.7 Non-direct Measurements

A substantial amount of data already exist for the outer continental shelf (OCS) Chukchi Sea as a result of extensive, multidisciplinary research programs (both industry and government) conducted over the past five years. These data are presented in the EMP Appendix A (OF, 2014) and will be used as part of the Phase I baseline characterization. Hence, baseline-characterization data already exist from empirical data collected in the past 5 years.

3.8 Data Management

During this study, both hard copy and electronic data records will be generated in the field by several principle investigators from different organizations. Each PI is responsible for ensuring that records generated by their field staff follow the requirements of this QAPP. Similarly, field samples will be analyzed in several fixed laboratories for a variety of parameters; the laboratory manager is responsible for ensuring that data management procedures are consistent with this QAPP. All the organizations that collect data in support of the EMP will maintain the original records that support their findings, report detailed results and provide copies of their records to OF at the end of each reporting phase.

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3.8.1 Document Control

The QAPP and associated SOPs are controlled documents. The following procedures will be implemented to ensure that project personnel have the current versions of these documents.

- The document version number and effective date is defined in the header control block.
- Each organization will maintain a master list of current SOPs, which are approved by management and assigned an effective date.
- A distribution list is maintained for this QAPP and associated SOPs.
- SOPs that describe environmental data collection activities must be reviewed prior to use to ensure they are current and updated as needed.

Field and laboratory logbooks must be bound, dated, paginated and distinctly labeled.

3.8.1.1 Field Documentation

Field records must provide a detailed description of sample collection activities to ensure that samples and data are traceable and defensible. FWS SOP-01 *Field Documentation* describes these procedures. Field observations will be documented in real time in bound field logbooks and will provide a record of field activities, observations, and measurements during sampling. All field records and documentation must comply with the documentation requirements defined in this QAPP. Field forms required for this study will be included in the EMP project Sampling and Analysis Plan. The field logbooks will be reviewed and approved independently at the end of each survey day.

3.8.1.2 Laboratory Documentation

Documentation of all laboratory activities is critical for tracking data and evaluating the success of any activity. Laboratory documentation requirements must be defined in a laboratory QA Manual or specific SOPs. At a minimum, the laboratories will maintain the following documentation records:

- Calibration and maintenance of all instruments and equipment involved in the collection of environmental data
- Preparation of calibration standards, spiking solutions, and dosing solutions such that each unique preparation can be tracked to the original (neat) material
- Lot numbers for all standards, stock solutions, reagents, and solvents
- Sample processing or preparation for testing such that it is traceable to sample receipt records
- Sample analyses and results of analyses
- Rejected data, accompanied by explanations of the failure and the corrective action
- Data reduction formulas such that reported data are traceable to raw data.

3.8.2 Data Storage Requirements

Storage of project data must ensure that the integrity and traceability of data are maintained. In the field, electronic data will be backed up daily to a second media (e.g., separate hard drive, thumb drive, CD),

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labeled, and stored securely. Field logs must maintain an inventory of the location of electronic files. Each laboratory must have a documented system of daily incremental and at least monthly full data backups, storage and recovery. Electronic data files received from the laboratories will be stored on a networked project folder with incremental daily and full monthly backups. Storage locations must be appropriate for the media (paper or electronic) and limit access or availability of the data. Once the study is complete, original field records and copies of laboratory records will be submitted to Shell. All hardcopy and electronic project files will be archived by the organization that generated the data for at least 5 years.

3.8.3 Documentation Standards

All data generated during the course of this project must be able to withstand challenges to their validity, accuracy, and legibility. To meet this objective, data are recorded in standardized formats and in accordance with prescribed procedures. The documentation of all environmental data collection activities must meet the following minimum requirements:

- Data must be documented directly, promptly, and legibly. All reported data must be uniquely traceable to the raw data. All data reduction formulas must be documented.
- Handwritten data must be recorded in ink. All original data records include, as appropriate, a description of the data collected, units of measurement, unique sample ID and station or location ID (if applicable), name (signature or initials) of the person collecting the data, and date of data collection.
- Any changes to the original (raw data) entry must not obscure the original entry. The change must be initialed and dated by the person making the change.
- The use of pencil, correction fluid, and erasable pen is prohibited.

During the project, records will be maintained in a secure location that minimizes loss as follows:

- Hardcopy records will be maintained in the project files of the principle investigator at each organization. This includes administrative records, field logs, and other field raw data. Field logs and custody forms will also be scanned and saved in pdf format and saved in a networked project folder.
- Electronic records will be maintained on in a networked project folder. No project files may be maintained on personal computers except as temporary working files.
- The results of QA/QC reviews, audits, and assessments will be saved in the QA network folder. Hardcopy audit records will be maintained in the QA files.

3.8.4 Hardware and Software Requirements

The following computer hardware and software standards requirements are established:

- Software used to capture or transfer data electronically from analytical instruments to data management systems must be described in QA documentation, and validated prior to use.

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- Data tracking from raw output to final values must be maintained. Data access and change control must be defined, limited, and traceable.
- Calculations performed by analytical instruments, data management software, and spreadsheets must be verified.
- All data and derived products will be stored in the laboratory computers and backed up on CDs.

3.8.5 Changes and Deviations

During the conduct of this study, it may be necessary to modify the planned activities. Modifications that are anticipated prior to field or laboratory work will be reported to the LS, who will assess the potential impact and contact the client if the changes are major (e.g., those that would affect the study objectives, design, or data quality). All modifications will be described in the final report.

Changes that are not anticipated prior to the planned activities are deviations and must be communicated and documented. Documentation will include an assessment of any impact that the deviation has on the study design and data quality, and any corrective action implemented. Minor deviations (e.g., those that would not impact the study objectives, design, or data quality) will be reported to and approved by the appropriate PI and the LS. Major deviations (e.g., those that could impact the study objectives, design, or data quality) will be reported to the LS and client. A discussion of major deviations and potential impact on the project objectives will be included in the final report.

3.8.6 Data Reporting

The reporting requirements for this project are defined by the analytical methods and intended use of the data.

Field measurements will be reported as follows:

- Water and sediment depth: feet
- Station location: WGS84 latitude and longitude (may change in field, system and units are always recorded with coordinates)

The concentrations of chemical compounds analyzed for this project will be reported in the units defined in Tables 3-10 through 3-15. Sediment concentrations will be reported on a dry weight basis and tissue concentrations will be reported on a dry weight basis. Additional data reporting requirements are as follows:

- Total PAH: the sum of all PAH compounds determined, including alkyl homologues, except retene (to ensure comparability to historical data)
- Total PAH16: the sum of the 16 priority pollutant PAHs (EPA method 610) Total TPH (SHC): the concentration based on the total resolved compounds and unresolved complex mixture in the SHC (F1) fraction (C9 – C40).
- Total SHC: the sum of the individual resolved SHC target compounds (C9 – C40)
- TAH: Method 602 volatiles +_o, m, p-xylene

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- TAqH: TAH+PAH (sum of the 16 PAH, as defined above)
- Non-detect values in sums: Sum of EPA 16 priority pollutant PAH (Total PAH16) will include all detected compounds plus all non-detected compounds using ½ of the MDL reported for each non-detected compound. All other summations will use a zero value for non-detects when used to calculate summations.

3.8.7 Data Reduction

Data reduction is the process of converting raw numbers (*e.g.*, numbers of organisms per replicate) into data that can be displayed graphically, summarized in tables, or compared statistically for differences between mean values for sampling times or stations. The data discussed in this section are those data that require some manipulation before being submitted to the data management consultant for entry into the database.

3.8.7.1 Infaunal Analysis

Historical and new data will be assessed for two attributes. These include standardizing the taxonomy to equivalent 'species' taxa groups and identification and removal of non-benthic species (e.g., pelagic organisms). All original data will be provided but the analyses to be performed will be based on these two modifications to the data.

3.8.7.2 Organics and Metals

GC/MS and GC/FID data will be acquired on PC-based Chemstation® minicomputers with dedicated chromatography software and reduced using MS Enviroquant® software. All GC/MS and GC/FID data files will be transferred electronically to a PC so that the data can be incorporated into an electronic database or spreadsheets for final quantification and tabular results presentation. Data for metals analysis by CVAF, CVAAS, ICP-MS, FAAS, and ZGFAAS are collected and processed by the instruments' software systems. Processed data are electronically transferred to Excel™ spreadsheet format for EDD generation. The final reduction of analytical chemistry data will account for the size of the processed sample and dilution factors.

3.8.7.3 Total Organic Carbon

Total organic carbon measurements are acquired on instrument software and downloaded onto Excel™ spreadsheets for final quantification and tabular results presentation. TOC results will be reported as percent total organic carbon on a dry weight basis.

3.8.7.4 Grain Size

Grain size will be reported as percent of the total for each size fraction measured. Silt content is determined by subtracting the total clay content from the mud content. Data are entered onto a spreadsheet for calculation of silt content. In addition to weight percent by size class, the Gravel: Sand: Silt: Clay ratio and a numerical approximation of mean size and sorting (standard deviation) are calculated.

3.8.7.5 Sediment Profile Imaging Analysis

After visual and computer image analyses are completed, a standard set of parameters (Table 3-15) taken from both analyses is combined and tabulated for reporting. If appropriate, statistical analyses can be done to test hypotheses by applying appropriate parametric (*e.g.*, *t*-test, ANOVA) and/or nonparametric techniques.

3.9 Data Review/Qualification

Data review conducted for this project is designed to ensure that reported data are accurate and traceable. Each organization generating data will perform the following data review:

- All data that are hand-entered from an original source to another location (e.g., from field logs to spreadsheets or from an instrument output to a spreadsheet) will be verified by the Lab Manager prior to use in calculations or entry into a database.
    - Manually transcribed data are highly susceptible to errors, which could include transposing numbers in the copying process, mis-associating data (e.g., assigning results to the wrong sample), and creating untraceable data by assumption, interpolation, extrapolation (e.g., adding information such as units, times or dates that are not recorded in the original entry). Hand-entered data must be verified 100%.
    - Electronically-transcribed data errors generally consist of an entire entry or record being copied into the wrong location or not copied over at all, resulting in missing data.
- All manual calculations will be performed by a second person to verify that calculations are accurate and appropriate.
- Calculations performed by software will be independently verified to ensure that the formulas are correct, appropriate, and consistent, and that calculations are accurately reported. .

Reported data will be independently reviewed to verify that reported data are accurate and traceable. The review will verify that data were generated according to the methods and MQC defined in this QAPP, including review of:

- Sample holding times and preservation requirements vs. the requirements of Table 3-6.
- Initial and continuing calibration methods, frequency and results vs. the requirements of Table 3-16.
- Sample extraction and analysis procedures vs. the requirements of Table 3-9.
- Laboratory QC results vs. the requirements of Table 3-20.

Laboratory data that do not meet the requirements of this QAPP will be re-extracted and/or re-analyzed whenever possible. However, if it is not possible (e.g., sample consumed) or beneficial (sample exceeds holding time) to re-extract or re-analyze then data qualifiers will be added and reported with the data. Laboratory data qualifiers are defined in Table 3-21.

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Table 3-20 Analytical Data Qualifiers

Laboratory Qualifiers[1]	
Organic Chemistry Data Qualifiers	
N	The QC result does not meet the accuracy, precision, or method blank MQO. The quality control result, not the related sample compound, is qualified.
n	The QC result does not meet the base accuracy or precision MQO, but meets the contingency criteria. Spiked sample (MS/MSD) and SRM result is less than 5X the native sample concentration or less than 5X the MDL (field sample replicate).
B	Blank contamination. The analyte was detected in the field sample at <5X the Method Blank concentration; the qualifier is applied to the field sample. The B qualifier is not applied if the sample data is J-qualified.
J	Estimated value: The analyte was positively identified but at a concentration less than the sample-specific RL.
U	Analyte was not detected at a 3-5:1 signal:noise ratio. The reported data value is the sample-specific MDL.
D	Dilution analysis value. The initial analysis was outside the calibration range of the instrument.
E	Estimated value. Result is greater than the highest calibration concentration.
ME	Estimated value. Significant matrix interference.
MI	Significant matrix interference. Value could not be determined or estimated.
T	Holding time (HT) exceeded.
H	Surrogate compound was diluted out. Qualifier used when surrogate recovery is affected by dilution of sample extract.
NA	Not applicable.
Metals Chemistry Qualifiers (including Mercury and Methylmercury)	
N	The QC result does not meet the accuracy, precision, or method blank MQO. The quality control result, not the related sample compound, is qualified.
J	Estimated value: The analyte was positively identified but at a concentration less than the sample-specific RL.
U	Not detected above the MDL: The associated reported value is the MDL, adjusted by any dilution factor used in the analysis (i.e., the sample-specific MDL).
D	Dilution analysis value. The initial analysis was outside the calibration range of the instrument.
NA	Not applicable
NS	Not spiked

Notes:

[1]MDL = method detection limit, as determined by EPA Method (US EPA 1997. 40 CFR part 136, Appendix B rev. 1.11)
RL = reporting limit, the field sample concentration equivalent to a final extract concentration of that of the low calibration standard
MRL = method reporting limit, the MDL multiplied by 3.18 (based on an MDL determination using 7 replicates).

As a final review, the project principle investigator or laboratory manager at each organization will review and make professional judgments about the usability of data generated by that organization based on the field notes, field and laboratory QC results¸ and environmental reasonableness.

Data reported in tables or deliverables will be audited by, or under the direction, of the Project QA Officer. Errors noted in data audits will be communicated to analysts and project management and corrected data will be verified. No data measurements will be eliminated from the reported data or database and data gaps will not be filled through interpolation, extrapolation, or with other existing data. The loss of samples during shipment or analysis will be documented in the data reports.

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4.0 REFERENCES

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