QUALITY ASSURANCE PROJECT PLAN ADDENDUM

Radiologically Impacted Material in Areas 1 and 2
West Lake Landfill Operable Unit -1
Bridgeton, Missouri

October, 2015
Prepared by S.S. Papadopulos & Associates, Inc.

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10/13/15

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DISTRIBUTION LIST

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List of Acronyms

% R - Percent Recovery
CEC - Cation-Exchange-Capacity
CSWP - Core Sampling (Phase 1B, 1C, and 2) Work Plan – Revision 1
DQO – Data Quality Objections
EMPS - Electron Microprobe Analysis
EMSI - Engineering Management Support Inc.
EPA - United States Environmental Protection Agency
FE – Feezor Engineering
g – grams
IDL – Instrument Detection Limit
kg - kilograms
L- Liters
LCS - Laboratory Control Samples
MCL – Materials & Chemistry Laboratory, Inc.
MDL – Method Detection Limit
meq - milliequivalents
mg – milligrams
MS – Matrix Spike
MSD – Matrix Spike Duplicate
MSW - Municipal Solid Waste
PARCCS - Precision, Accuracy, Representativeness, Comparability, Completeness, & Sensitivity
pCi – picocuires
QAPP – Quality Assurance Project Plan
QA/QC - Quality Assurance/Quality Control
RIM – Radiologically Impacted Material
RL – Reporting Limit
RPD – Relative Percent Difference
SBLT - Sequential Batch Leaching Tests
SPLP - Synthetic Precipitation Leaching Procedure
SSP&A - S.S Papadopulos & Associates
TCLP - Toxic Characteristic Leaching Potential
TOC – Total Organic Carbon
wt% - Percentage by weight
XRD – X-ray diffraction

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Section 1
Project/Task Organization

The organization and management of the project is detailed in the January 8, 2014 Core Sampling (Phase 1B, 1C, and 2) Work Plan – Revision 1 (CSWP) prepared by Feezor Engineering., Inc. (FE). S.S. Papadopulos & Associates, Inc. (SSP&A) has been retained to assist Engineering Management Support Inc. (EMSI) in the fate and tran sport evaluations at the site as requested by the United States Environmental Protection Agency (EPA).

Section 2
Problem Definition/Background

West Lake Landfill contains, in places, radiologically impacted material that is referred to as RIM. Areas 1 and 2 of West Lake Landfill are identified by the EPA as Operable Unit 1 of the West Lake Landfill Superfund Site. Remedial actions to address RIM are being directed by EPA.

2.1 Project/Task Description

As part of the remedial actions directed by the EPA to address RIM, EMSI submitted a work plan (Work Plan) for additional characterization efforts of West Lake Landfill Areas 1 and 2 in a letter dated July 6, 2015. The Work Plan was based on, and incorporated procedures from other work plans previously submitted to, and approved by, the EPA. The previously submitted work plans (i.e. CSWP) included the various requirements of a quality assurance project plan (QAPP) for sample collection and plan specific measurements and analyses. The scope of work discussed in the Work Plan that will be covered by this QAPP addendum includes the additional laboratory analytical testing to obtain site-specific data for use in the fate and transport evaluations requested by the EPA.

As discussed in the Work Plan, the additional testing is designed to identify and distinguish the chemical composition of the materials containing radionuclides and the speciation of the radionuclides in these materials, and to provide data to parameterize the geochemical fate and transport model. Specifically, two samples will be collected from each of four borings in Area 1, and two samples from each of six borings in Area 2 (resulting in a total of 20 solid samples). The first sample obtained from each boring will be selected from a depth interval that displays high gamma readings (based on the gamma scans of the core samples). Analytical data from these samples will be used to evaluate the geochemistry, stability and leachability of any radionuclide occurrences in Areas 1 and 2. The second sample will be collected from a deeper interval that does not display elevated gamma readings. Analytical data from these samples will be used to evaluate potential attenuation of radionuclides that may be mobilized from the overlying RIM. Samples will be placed in plastic bags, vacuum-sealed, and subsequently shipped to the laboratory on ice in order to preserve the in-situ chemical oxidation state of the samples. Also, prior to analysis, samples will be air-dried and homogenized by the laboratory in a glove box.

Table 1 presents both a summary of the proposed laboratory analyses to be performed in support of the fate and transport evaluations and the intended use of the data from each of the tests. Samples to be tested for fate and transport-related parameters will be subject to the following analyses:

- Uranium, thorium, and radium isotopes;

- Major cations and anions (including calcium, magnesium, sodium, potassium, barium, carbonate, sulfate, fluoride and phosphate);

- pH and redox indicators (Fe(II), Fe(III), sulfide, and U(VI));

- Total organic carbon (TOC), whi ch assesses the levels of humic and fulvic acids that affect partitioning and mobility of radionuclides (and the longevity of potentially-reducing conditions within the landfill);

- X-Ray Diffraction (XRD), which quantifies the abundance of major minerals (e.g. barite and/or calcite in the waste) that potentially-affect lea chate composition and radionuclide speciation (XRD provides a semi-quantitative description of the primary minerals present in a sa mple to corroborate the calcula ted mineralogy based on cation and anion analyses);

- Sequential extraction analysis, which consists of sample digest ion in a series of sequential extraction steps designed to dissolve specific miner als, will be used to access the speciation of radionuclides in the samples.

- Scanning Electron Microscope with Energy Dispersive x-ray Spectrometry (SEM/EDS), which provides a semi -quantitative method for elemen tal mapping (e.g., barite, gypsum, calcite, and oxides);

- Cation-Exchange-Capacity (CEC), w hich estimates the potential c apacity of the waste/soil to adsorb radionuclides; and,

- Sequential batch leaching tests (SBLT), which will primarily be used to evaluate the parameterization of the fate and transport model by comparing measured and simulated SBLT results.

Table 1. Target analytes, methods, and reporting limits for soil samples and aqueous extractions

Analysis	Parameter	Method Reference	Soil Samples		Aqueous Extractions	
			Units	Reporting Limit	Units	Reporting Limit
Radionuclides	Ra-226	EPA 903.0 Modified	pCi/g	1	pCi/L	1000
	Ra-228	EPA 904.0	pCi/g	1	--	--
	Th-230	HASL EML Th-01 Modified	pCi/g	1	--	--
	Th-232	HASL EML Th-01 Modified	pCi/g	1	--	--
	U-234	EPA 6020	mg/Kg	0.001	--	--
	U-235	EPA 6020	mg/Kg	0.01	--	--
	U-238	EPA 6020	mg/Kg	1	--	--
	Total Radium	***	--	--	pCi/L *	1000
	Total Thorium	HASL EML Th-01 Modified	--	--	pCi/L *	1000
	Total Uranium	EPA 6020	--	--	mg/L *	1
Major Cations and Anions	Barium	EPA 3050, EPA 6010	mg/Kg	2	mg/L *	0.2
	Calcium	EPA 3050, EPA 6010	mg/Kg	20	mg/L *	5
	Carbonate	Water Leach, SM 2320E	mg/Kg	per method	mg/L	per method
	Fluoride	Water Leach, EPA 300	mg/Kg	2	--	--
	Iron	EPA 3050, EPA 6010	mg/Kg	20	mg/L *	5
	Magnesium	EPA 3050, EPA 6010	mg/Kg	20	mg/L	5
	Manganese	EPA 3050, EPA 6010	mg/Kg	20	mg/L *	0.2
	Phosphate	Water Leach, EPA 300	mg/Kg	12	--	--
	Potassium	EPA 3050, EPA 6010	mg/Kg	20	mg/L	5
	Sodium	EPA 3050, EPA 6010	mg/Kg	20	mg/L	5
	Sulfate	Water Leach, EPA 300	mg/Kg	12	mg/L *	60
Redox Indicators	Ferric Iron	EPA 6010 by difference with Ferrous	mg/Kg	TBD	--	--
	Ferrous Iron	SM 3500-Fe B	mg/Kg	TBD	--	--
	Sulfide	EPA 6010 / EPA-OW-OST 376.3 **	mg/Kg	TBD	--	--
	U(VI)	MCL-7737	mg/Kg	TBD	--	--
General Chemistry	Cation Exchange Capacity	EPA 9081	meq/100g	per method	--	--
	Total Organic Carbon	EPA 9060/9060A	wt. %	0.01	mg/L *	1
	pH	EPA 9045D	std	0.05	std	0.05
Non-Destructive Testing	X-Ray Diffraction	MCL-7708	wt. %	3-5	--	--
	SEM/EDS	MCL-7712	--	--	--	--

"--" = Not Requested or Not Applicable
TBD = To be determined, dependent on the spike recovery in the sample
per method = the reporting limit is dependent on the nature of the sample (carbonate on the pH; CEC depends on cation concentr ations)
* Calculated based on Sequential Extraction Procedure Step 1
** Sulfide determination will be difference between total sulfur by EPA 3050 / 6010 and preparation for acid volatile sulfur EPA 376.3 / EPA 6010
*** Total Radium will be determined by summing the results from Radium-226 and Radium-228.

Section 3
Quality Objectives and Criteria

Data quality objectives (DQOs) are qualitative and quantitative statements that define the type, quality, and quantity of environmental data appropriate for the intended application. The overall DQO for this project is the obtain data of sufficient quality to allow for the evaluation of the fate and transport of site contaminates of concerns, primarily RIM.

The primary procedural tools required for achieving these DQOs are:

- Application of analytical methods with detection limits suitable to meet this DQO;
- Sampling methodologies to prevent cross-contamination and sample degradation;
- Collection of suitable laboratory and field duplicates and blanks; and
- Decontamination methods to prevent cross-contamination of samples.

Analytical performance requirements for this work are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS).

3.1 Precision

Precision is a measurement of the degree of agreement of replicate data, which is quantitatively assessed based on the relative percent difference. The relative percent difference (RPD) is calculated with the following equation:

$$RPD = \frac{A - B}{\frac{A + B}{2}} \times 100$$

where:
A = The larger of the two values.
B = The smaller of the two values.

For this project, generalized acceptance criteria of 20% RPD and 35% RPD will be used for water and soil samples, respectively. Failure to achieve a value below these levels will require a review of the field and laboratory documentation for these samples to assess possible causes, and may result in corrective actions.

3.1.2 Field Precision

Field precision will be assessed through the collection and measurement of field duplicate samples. Duplicate samples will be analyzed for a minimum of 5% of samples to check for overall variability introduced by sample heterogeneity, sampling and analytical procedures.

3.1.3 Laboratory Precision

Laboratory precision is assessed through the calculation of relative percent differences (RPDs) for two replicate samples. The precision of the analysis can be inferred through the use of

one of the following: laboratory control duplicate samples; matrix spike and matrix spike duplicate (MS/MSD) samples, or unspiked duplicate samples. The laboratory analyzes one or more of these duplicate samples at a standard rate per type of analysis, depending upon the analytical method.

3.2 Accuracy

Accuracy is the degree of agreement between a measurement or observation and an accepted value.

3.2.1 Field Accuracy

Field accuracy is assessed through the adherence to all sampling handling, preservation, and holding time requirements, use of appropriate field equipment and blanks. Field blanks for soil and groundwater samples will be analyzed to check for procedural contamination that may cause sample contamination. Accuracy of field instruments will be assessed by daily instrument calibration and calibration checks. These calibration checks will be documented in field notes.

3.2.2 Laboratory Accuracy

Laboratory accuracy is assessed by the analysis of matrix spikes (MS) and laboratory control samples (LCS). The results are expressed as a percent recovery. Surrogate recoveries may also be used to assess accuracy. Method blanks are used to assess contamination resulting from laboratory procedures. Laboratory control samples, method blanks, and preparation blanks will be analyzed at a standard frequency per type of analysis.

The percent recovery (% R) is calculated with the following equation:

$$\% R = \frac{A - B}{C} x\ 100$$

where:

A = The analyte concentration determined experimentally from the spiked sample.
B = The background level determined by a separate analysis of the unspiked sample.
C = The amount of the spike added.

3.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic environmental condition. Representativeness is a subjective parameter and is used to evaluate the efficacy of the sampling plan design. Representativeness is demonstrated by providing full descriptions of the sampling techniques and the rationale used for selecting sampling locations in the project planning documents. The measure of representativeness is answered during the preparation of the sampling and analysis approach and rationale, and then reassessed during the data usability process.

The sampling and analysis approach and rationale are defined in the Work Plan and in the CSWP.

3.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was planned to be obtained under normal conditions. Percent completeness is calculated with the following equation:

$$\%\ Completeness = \frac{Valid\ Data\ Obtained}{Total\ Data\ Planned} \times 100$$

Experience on similar projects has shown a reasonable goal considering laboratory performance is 80 percent completeness. If sufficient valid data are not obtained, corrective action will be initiated by the Project Manager to modify field, data shipping, or laboratory procedures.

3.5 Comparability

Comparability expresses the confidence with which one data set can be compared with another data set obtained during parallel or previous investigations. Comparability can be related to precision and accuracy as these parameters are measures of data reliability.

Chemical samples from the same media are generally considered comparable if the same procedures for collecting and analyzing the samples are used, if the samples comply with the same Quality Assurance/Quality Control (QA/QC) procedures, and if the units of measurement are the same. Comparability in this project is addressed through the use of uniform analytical methods, sampling procedures and field procedures across the entire site.

3.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method is commonly referred to as the detection limit. Although there is no single definition of this term, the following terms and definition of detection limits will be used:

- Instrument detection limit (IDL) is the minimum concentration that can be measured from instrument background noise under ideal conditions.

- Method detection limit (MDL) is a statistically determined concentration. It is the minimum concentration of an analyte that can be measured and re ported with 99 percent confidence that the analyte concentration is greater than zero as determined in the same or a similar matrix. Because of the lack of analytical precision at this range, sample results (if reported by the laboratory) greater than the MDL but less than the reporting limit (RL) would be qualified as "estimated".
- Reporting limit (RL) is the concen tration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operati ng conditions. This value is v ariable and highly matrix dependent. It is the minimum concentration that will be report ed as unqualified by the laboratory.

For sensitivity, the quality objective is to analyze data with a method that achieves RLs that are below or equal to the task-specific target analysis go als or concentrations. Planned sensitives are presented in Table 1.

Section 4
Special Training/Certification

As discussed in the approved CSWP quality assurance section, th ere are no special training/certifications required for this scope of work.

Section 5
Documentation and Records

Documentation and records are detailed in Section 9 of the approved CSWP.

Section 6
Sampling Process and Design

Specific methods and designs for s ample collection and analysis , handling, reporting and other aspects of sample collection are detailed in Section 4 of the approved CSWP.

Section 7
Sample Handling and Custody

Specific methods and designs for sample handling and custody are detailed in Section 4 of the approved CSWP.

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Section 8
Analytical Methods

This section provides information on the analytical methods for each medium of concern. In general, all analyses will utilize EPA-approved methods or other recognized standard methods. Method references for laboratory analyses that may be performed for the anticipated work are provided in Table 1. Sample size, preservation requirements and holding times for analytical parameters not detailed in the CSWP are summarized in Table 2.

The primary laboratory for this study will be Materials & Chemistry Laboratory, Inc. (MCL) of Oakridge, TN. The primary laboratory's Quality Assurance Manual, and internal analytical Standard Operating Procedures are attached as Appendix 1.

Laboratory turnaround times for this project shall generally range between 8 and 12 weeks.

Table 2. Sample requirements and holding times for solid samples

Sample Type	Mass Requested (grams)	Preservation	Hold Time (days)	Container
Radiological	250	<6°C	180	Poly Bottle or Bag
Non-Radiological	1000	<6°C	180	Poly Bottle or Bag

Section 9
Quality Control

Quality control is discussed in Section 4.9 of the approved CSWP.

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Section 10
Data Management

Data Management is discussed in Section 9 of the approved CSWP.

Section 11
Assessment and Response Actions

This section presents the internal and external checks (assessm ents) that have been built into this project to assure that:

- Elements of this QAPP addendum have been correctly implemented as prescribed for all tasks this project;
- The quality of the data generated is adequate and satisfies the DQOs that have been identified in this QAPP addendum; and
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

Assessment activities may include surveillance, inspection, pee r review, management systems review, readiness review , technical systems audit, perf ormance evaluation, and data quality assessment. Assessment and response actions are detailed in the approved CSWP.

Section 12
Data Review, Verification, Validation, Usability & Reconciliation

This section provides a description of the QA activities that will occur after the data collection phase of the project is completed. Implementation of this section will determine whether or not the data conform to the specified criteria, thus satisfying the project objectives.

Data Review, Verification, Validation, Usability & Reconciliation is discussed in Section 8.3 of the approved CSWP.

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ATTACHMENT 1

MATERIALS & CHEMISTRY LABORATORY, INC. (MCL) QUALITY ASSURANCE PLAN

MATERIALS & CHEMISTRY LABORATORY, INC.

"Linking Technology to Solutions"

Quality Assurance Plan
MCL-7701

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park
Building K-1006
2010 Highway 58, Suite 1000
Oak Ridge, Tennessee 37830

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1.0 ORGANIZATION

1.1 Introduction

Materials and Chemistry Laboratory, Inc. (MCLinc) provides technical support to a variety of customers and programs. Work done may be classified at levels up to U.S. Department of Energy (DOE) "Q" or "QX" (S-RD [level: secret – Category: Restricted Data]), and may involve radioactive, special nuclear materials (SNM), and/or hazardous materials. Scope of work includes, but is not limited to, characterization studies, research projects, development efforts, lab-to-bench-to-pilot scale processes, process optimization, and methodology development. Quality is inherent in all aspects of MCLinc work. This plan and the references herein, ensure that a management framework is defined for the establishment of quality MCLinc practices.

It is noted that this plan does not specifically address all aspects of the Industrial Hygiene Analysis Laboratory (IHL) within MCLinc. The IHL is an American Industrial Hygiene Association Program (AIHA) Laboratory Accreditation Program (LAP), LLC (AIHA) accredited laboratory. The latest AIHA assessment was done under the requirements of International Organization for Standardization/International Engineering Consortium (ISO/IEC) 17025 international standard. The IHL operates under a stand-alone quality plan, MCLinc, "Industrial Hygiene Laboratory Quality Assurance Manual," MCL-7719. The information contained within the MCLinc Quality Assurance Plan (QAP) will still apply to the overall operation of all IHL functions, but will not specifically address some of the AIHA-required details that are unique to the IHL. This document and other supporting Standard Operating Procedures (SOPs) will apply to all MCLinc AIHA accreditations.

1.2 Quality Assurance Policy

The MCLinc Quality Assurance (QA) Policy approved by the MCLinc President is to assure that the *QA* practices utilized by the MCLinc staff conform to requirements, standards, and responsibilities necessary for maintaining a quality organization in conjunction with DOE, and customer-based expectations. This policy incorporated into this QAP will help to minimize the risk and environmental impact of processes influenced and performed by MCLinc as well as maximizing the safety, reliability and performance of MCLinc methodologies and practices. The MCLinc QA Policy must be

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rigid to assure quality objectives are met, but also dynamic by having procedures in place to allow continual improvement in the quality management process (annual assessment, SOP change procedure, and corrective action procedures are examples of means to allow improvement).

This QAP is designed to specifically meet the management and technical requirements for testing facilities of the internationally recognized standard ISO/IEC 17025 and American Society of Mechanical Engineer, Nuclear Quality Assurance, Level 1 (ASME NQA-1), National Environmental Laboratory Accreditation Conference related U.S. Environmental Protection Agency (EPA) and DOE documents noted in the Reference Section. See Appendix A for a cross-reference table, by section of this QAP, to the specific requirements of ISO/IEC 17025, Title 10, Code of Federal Regulations (10 CFR) Part 830.120, American National Standards Institute/American Society for Quality Control (ANSI/ASQC) E4-1994, and ASME NQA-1. Changes to this document may only be made with the approval of the MCLinc President and Quality Assurance Officer (QAO) per the SOP "Document Control", MCL-7703.

1.3 Organizational Responsibilities

The organizational structure of MCLinc is shown in Appendix B. MCLinc must be able to maintain flexible work responsibilities to ensure that a wide variety of customer, Site (East Tennessee Technology Park and URS/CH2M Oak Ridge, LLC and Landlord (DOE and Community Reuse Organization of East Tennessee [CROET]) requirements can be met.

1.4 Functional Responsibilities

The MCLinc President provides daily guidance and administrative support to the MCLinc staff and is committed to ensuring compliance to this QAP and ISO/IEC Standard 17025, AIHA, and other quality requirements of our customers. The MCLinc President is supported by the Technical Director (TD) and the Laboratory Manager (LM). These positions provide routine assistance to personnel and customers on the capabilities and application of MCLinc resources to solving problems.

The TD has responsibility to provide technical direction to the Project Manager (PM) and technical assistance to our clients. The LM has responsibilities for the day-to-day operation of the laboratories and to make sure resources are available to meet the needs of our clients and this plan. The Quality Assurance Specialist (QAS) performs quality duties as assigned by the QAO. The QAS is the designated alternate for the QAO in his absence.

All staff members of MCLinc are responsible for ensuring that customer objectives (i.e., quality, time frame, budget, applicability) are met in accordance with this plan and any other applicable documentation. The work performed by any and all staff members is necessary to meet our management quality objectives and those of our clients. A staff

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member is empowered to stop work due to any safety issue or when the quality of the product is endangered and report such concerns to the QAO. Appendix C lists MCLinc personnel and their support functions which will help ensure the quality objectives of MCLinc and its customers are met. The QAO is the point of contact for the implementation and enforcement of quality-based procedures. Conflicts in operating methods and procedures will be resolved by the QAO with support from the appropriate management personnel. The QAO is committed to compliance to this QAP and ISO/IEC Standard 17025 and other quality requirements of our customers. The QAO, as necessary, develops and issues SOPs or QA Directives in memo format to further define or explain items covered by this QAP or other areas needing procedures defined.

The QAO has the authority to stop work at any time to assess a reported problem or investigate a quality system failure or trend.

The PM is either self-appointed or is selected by the LM or MCLinc President based upon the nature of the project. The PM is the person responsible for the control and coordination of all activities associated with the successful completion of a customer task.

1.5 Facility/Security Attributes

MCLinc is leasing the K-1006 facility from the DOE, through CROET as part of the DOE reindustrialization initiative. The brick facility is approximately 28,000 square feet (ft^2). The area available to MCLinc under its lease with the CROET is approximately 25,300 ft^2. The facility is designed to be a laboratory facility. The second floor contains office space (15 rooms) and one storage closet. The first floor accommodates approximately: 30 labs, 12 offices, 5 administrative/common areas, 2 maintenance areas, 5 hallways, and 2 utility chases. There is approximately 12,445 ft^2 of laboratory space and 12,885 ft^2 of non-laboratory space within the MCLinc facility. The facility is dedicated to handling virtually any type of environmental contaminant and is operated by a multidisciplinary staff qualified to address technical issues pertaining to radiological and hazardous materials.

Although MCLinc is a private commercial entity, it still has some operational restrictions based upon where and what type of business it does. As part of the basis for MCLinc to continue to be authorized to perform classified work, a graded approach to physical security had to be implemented. The physical security, and hence the main basis for the security infrastructure of the MCLinc facility, is based upon three to four layers of access control. The first layer is that access to the site is monitored by Site Security with all visitors required to go through the security office. The second layer is the controlled access requirements (Hirsch badge reader system or controlled access key) to gain access into building K-1006. The third layer is controlled key access into laboratory areas. The fourth layer is the controlled access storage area within various laboratories. The Facility Security Officer (FSO) will assign keys. All assigned keys will either be physically controlled by the individual person or will be controlled by that individual using a unique

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lock/combination controlled storage area. Lost keys will be reported to the FSO immediately. This graded approach to physical security provides the required control over facility access for security issues as well as for proper chain of custody (COC) of certain materials and samples.

1.6 Commitment to Quality

The managers, owners and employees of MCLinc, an employee-owned company, are committed to a policy whereby all personnel are free of any undue internal or external commercial, financial, or other pressures and influences which may adversely affect the quality of the work. Any staff member feeling such pressures shall report this concern immediately to the LM, TD, QAO or the MCLinc President for investigation and corrective action.

As noted under the Facilities Section, the facility is secure and the staff is knowledgeable in the handling of confidential information for the DOE. This same approach is extended to all clients in that client confidential information or proprietary rights are maintained in confidence and all such documents or electronic files are stored in locked files or in computers password-protected and accessible only to authorized staff. See Section 5.5, Classified Materials. The MCLinc reputation and success depends on the high integrity of the staff. MCLinc's policy is that technical and business competence, impartiality, judgment, and data quality and operational integrity must be maintained at all times. These elements are key to maintaining the quality of our efforts. The employees therefore must be aware of their contributions to maintaining the management quality system.

The MCLinc management staff has the responsibility and authority to provide the resources to complete the above and through staff and project meetings and other communication tools (i.e., e-mail) encourage the staff to communicate their assessment of the management system. The MCLinc management also has the responsibility for training, implementation, maintenance and improvement of the management system and to identify and correct variances from the system. The QAO, TD, LM, PM and QA assessments are key in identifying any variance from this policy which must be investigated and corrective action taken including disciplinary action.

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2.0 MANAGEMENT SYSTEMS

2.1 Standards and Reference Materials

MCLinc has a need for a variety of standards and reference materials. Where possible, these standards and reference materials must be purchased from an ISO/IEC 17025; 2005 certified vendor. Traceability of these standards must also be demonstrated on the Certificate of Analyses of the standard. The standards and reference material must be handled, stored and used per the manufacturer's specification to avoid contamination and deterioration.

Many standard methods require use of second source standards to check primary standards (i.e., organics and metals). The "Quality Systems for Analytical Services" (QSAS) requires radiation calibration standards to be verified prior to use and annually as follows:

- At least three verification measurements of a standard are used to determine the mean value and standard deviation of verification results.
- Mean value is within 5 percent (%) of certified value.
- Two sigma deviations is less than 10% of the mean value.

If specifications are not met, corrective actions must be evaluated and implemented.

2.2 Calibration

Instrument and equipment performance evaluation, maintenance, and documentation are the responsibility of the instrument owner. Appendix D lists the responsible owner and authorized operator for the major instrumentation with the MCLinc facility. These instruments have specific QA documents that outline the minimum calibration requirements. For the general or common data acquisition laboratory equipment (e.g., balances, pH meters,) the "Calibration, Maintenance and Inspection Plan," MCL-7711, outlines the calibration and documentation requirements for those components which may influence the work being performed. When there is a need for outside calibration of laboratory equipment, the vendor/material used must be traceable to national standard setting bodies such as National Institute of Standards and Technology (NIST) or ISO approved.

2.3 Facility Maintenance

The housekeeping and maintenance of each MCLinc office or laboratory facility is the direct responsibility of all MCLinc personnel. MCLinc facilities should be kept clean and orderly and the temperature and humidity controlled to meet the needs of the testing instrument. If the MCLinc staff member responsible for an area is temporarily or permanently unable to comply with this standard, he or she should advise management of the problem. Specific health and safety requirements are outlined in the "Chemical Hygiene Plan," MCL-7702 and the "Health and Safety Plan," MCL-7717. Specific requirements for maintenance and facility documentation are provided in the "Calibration, Maintenance and Inspection Plan," MCL-7711.

2.4 Work Environment

MCLinc maintains a safe and clean working environment. All laboratory areas and materials are maintained in a clean and orderly fashion to ensure the work performed will not be compromised by the local environment of the laboratory facility. The MCLinc personnel performing work are responsible for ensuring that all cleanliness requirements are met prior to commencing work. The "Chemical Hygiene Plan," MCL-7702, provides additional detail.

2.5 Laboratory Supplies

These materials are stored and controlled based upon the hazardous nature of the material. Individual personnel are responsible for ensuring that the integrity of the laboratory supplies is adequate to meet MCLinc and the customer's expectations. The ordering, reporting and tracking of chemicals is addressed in the "Chemical Hygiene Plan," MCL-7702 and the "Procurement Control Plan," MCL-7727. The ordering, reporting, and tracking of radiological materials are addressed in the "Implementation SOP for the Radiation Protection Plan," MCL-7715.

2.6 Special Nuclear Materials (SNM)

The control and monitoring of SNM is detailed in "Nuclear Materials Control and Accountability Plan," MCL-7706. The Radiological Safety Officer (RSO) is responsible for oversight and control of SNM.

2.7 Material and Sample Receipt

Samples and materials are received at the MCLinc facility from various sources and require various levels of oversight and control. Sample login, tracking, documentation, archival, disposal, and/or return procedures are detailed in "Project Management Plan," MCL-7704 and Operator Aid Appendix O in MCL-7756, "Operator Aids." This procedure provides guidance on issues such as non-conformance reports (NCR), cross contamination, inspection log sheets, sample tracking and management, and laboratory records associated with sample management. The "Procurement Control Plan," MCL-

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7727 and Section 3.1 provide details on the quality control (QC) checks and documentation required for receipt of materials.

2.8 Controlled Samples

Controlled samples have COC documentation. COC forms can either be supplied by the customer or by MCLinc. The COC ensures that the samples will be maintained within the MCLinc facility. The COC form will be documented to reflect when the samples either leave the MCLinc facility or a non-MCLinc employee is provided direct, unsupervised, access to the samples. No internal COC control is required for samples remaining within the MCLinc facility.

2.9 Non-COC Samples

Many projects that are performed by MCLinc are on samples that do not have an associated COC and are typically representative of a process or condition that does not require COC control. These samples are maintained within the secure MCLinc facility. The use and control of these samples is the responsibility of the PM. The PM may elect to document any special handling or storage protocols that should be used for a given sample or group of samples. The PM is responsible for ensuring that proper documentation and labeling is provided to ensure that any MCLinc employee that may need to utilize these materials understands the specific requirements associated with the samples.

2.10 Sampling and Sample Preparation

In MCLinc projects where actual sampling of the process is required, the details of the sampling process and procedures to follow must be defined in a project sampling plan or scope of work. All samplers must be trained on the procedures and understand the critical importance of the sample to the project. The resultant sample must represent the source being sampled and the PM must define steps to be taken to best approximate a homogenized sample. This is also a critical step in sub-sampling samples received at the laboratory for testing.

The staff member responsible for the analysis shall determine sample preparation techniques utilized. Sample preparation techniques shall be documented. Good laboratory notebook protocols will be used when documenting the laboratory, data, and/or project activities. Additional quality, safety, and environmental aspects of sample preparation are provided in, "Sample Preparation Plan," MCL-7710.

2.11 Instrumentation and Maintenance

MCLinc has a variety of laboratory instrumentation ranging from very complex (e.g., transmission electron microscope) to standard laboratory instrumentation (e.g., pH meter). The level of the documentation required for the standardized use of instrumentation is decided by the LM. The instrumentation listed in Appendix D requires

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some level of QA and/or operational guidance. The use and control of the general laboratory equipment is provided in "Calibration, Maintenance and Inspection Plan," MCL-7711, technology specific SOPs (e.g., MCL-7708, "Electron Microscope Operations Plan"), project specific QA plans, vendor manuals, and other customer specific documentation. MCLinc staff members using an instrument are responsible for documenting non-routine maintenance and repairs, and maintaining an inventory of consumables and commonly needed parts.

2.12 Quality Control Samples and Assessment of Data

Since the vast majority of the projects performed by MCLinc are non-routine or the application of a routine procedure to a non-routine use, the measurement quality objectives vary significantly. The basic objective of all MCLinc measurements/analyses are to assure: (1) the procedure measures the parameter of interest, (2) the instrument/system is calibrated and operating properly, (3) the sample was properly prepared and handled in a way to minimize contamination, and (4) the data is calculated, reviewed and reported properly. Depending on the procedure or technique utilized MCLinc achieves the above by using QC samples. These QC samples include one or more of the following:

- Method Blank
- Instrument Blank
- Calibration Check Standard
- Laboratory Control Sample's (Duplicates)
- Matrix Spike and/or Matrix Spike Duplicate
- Duplicate Sample
- Certified Sample

In many cases the QC sample requirements, if not defined by the procedure, are defined by the client and MCLinc at time of project inception. Any anomalies or failures of QC samples must be evaluated and if persistent reported as a non-conformance requiring corrective action.

The laboratory control samples (LCSs) shall be used by the analyst to evaluate method performance. In cases where the method is run infrequently (less than 20 samples per month), the analyst shall evaluate LCS recoveries against criteria in the method or use a default of 100% ± 25% recovery. Corrective action will include rerun of the LCS and if it still fails evaluation by the QA*O* verses client project needs.

For analytical methods requiring LCSs and run frequently (more than 20 samples per month), LCS data shall be tabulated for review by the analyst to see trends with calculation of the standard deviation (sigma). The data points generated for each sample set should be evaluated as follows:

± 1 Sigma shall contain 2/3 of the points
± 2 Sigma shall contain 19/20 filter points

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± 3 Sigma shall contain ALL of the points

If not, corrective action as noted above shall be taken.

Shewhart type control charts may also be used to display and assess the data. These are especially useful for single analyte analyses.

The QA*O* will maintain a list of those methods using control tables or charts.

If required by the project the results of the QC samples may be utilized to calculate and estimation of uncertainty for the reported data in "Estimation of Uncertainty of Measurement (EUM)", MCL-7735.

2.13 Data Review and Evaluation (Design Control)

The PM will define the level of data review required to meet the project quality objectives and customer's expectations. The MCLinc minimum standard for data review is a two level review of an initial (level one) review by the analyst/instrument operator assuring that the analyses was properly performed, calculations are checked and the acceptance criteria for the method were met. The QAO, another analyst or TD will perform a second review (level 2 review) of the parameters listed plus the final report.

During the data review and quality analysis process, any quality data outside of method or project set criteria must be evaluated by the reviewer to determine appropriate corrective action. If the problem is caused by a systematic error, an investigation shall be performed to assure the proper corrective action is established. This approach is necessary to avoid reporting incorrect data to the client.

Additional internal data review (level three) will be provided by appropriate senior technical staff as warranted per the subject matter of the data and/or the requirements of the project. In no instance will data be reported without review. Information reported prior to completion of the review/evaluation process must be clearly identified as "preliminary data".

Computer programs that are used to produce test data or calculate test data must be self-checking or verified per "Verification of Data Software," MCL-7728.

2.14 Standard Operating Procedures (SOPs)

MCLinc uses SOPs to define routine analytical methods, quality systems, chemical hygiene, health and safety, security, and radiological. For simpler routine procedures, QC, or project specific requirements, MCLinc uses operator aids that are incorporated as controlled documents in MCLinc SOP MCL-7756, "Operator Aids," or as an attachment to an SOP.

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The key elements of SOPs and Operator Aids are presented in Table 2.14. Any deviations from these elements must be approved by QA.

Current routine procedures to be covered by this document, operator aids or SOPs include the following:

- Reagent water preparation
- Sample receiving
- Balance checks
- Preparation of standards
- Temperature monitoring of ovens and refrigerators
- Calibration of thermometers
- Preventative maintenance
- Calibration of mechanical pipettes
- Checking of hood velocities
- Detection limits studies
- Preventive maintenance
- Inspection of glove boxes
- Assessment of data

A complete list of SOPs and Operator Aids is found on the MCLinc Controlled Document Status List maintained by the Document Control Coordinator (DCC).

Table 2.14 Key Elements of SOPs and Operator Aids

Section	SOP*	Operator Aid**
Title	Cover Usage in a complete statement referencing a regulatory procedure as appropriate	Clear, simple statement
Purpose	Purpose may be detailed	Included in scope
Scope	Covers use and application	Simple description of purpose and scope
Responsibilities	Defines roles of analyst, supervisor, QA and management	Not applicable
Definitions	Provide any definitions unique to SOP	Define unique terms when used
Reagents	Define each with all the details (i.e., chemical name, formula, % purity, manufacture).	Define in procedure- reagents used and source only if unique
Standards	Define each including concentration or purity	Define in procedure
Equipment	Define equipment or instrumentation utilized	State in procedure equipment used but provide details only on specialized equipment
Reagents Preparation	Describe details of preparing reagents listed above	Include for non-routine reagents in the procedure

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Section	SOP*	Operator Aid**
Standardization and Calibration	Define process (including initial preparation of standards), standard numbers, levels, acceptance criteria, etc.	Cover in the procedure in stepwise format
Procedure	Stepwise details of the process which in some cases may include the why or background for each step	Simple steps to follow assuming a trained analyst
Safety (including any waste issues)	May be separate section or defined as necessary in SOP	Define in procedure where appropriate
Calculations	Outline information required and formula to use	Define in procedure the formula and its elements
QC	Define the types of QC samples required and the appropriate criteria for evaluating data. Define corrective action	Define in procedure QC samples required, corrective action, and define on prep sheet QC criteria
Documentation	Define preparation sheet or where data should be documented (i.e., notebooks)	Includes preparation sheet or defines what to record in notebook
Pollution Prevention and Waste Management	In all SOPs dealing with chemicals	Not usually needed
References	List applicable documents	List Documents
Other sections or attachments may be required to meet the needs of the SOP usage	Comment – It is also appropriate to have the SOP summarized as an Operator Aid and enclosed as an attachment.	Rarely needed

* These are the requirements for a SOP covering an analytical procedure. Other administrative or policy SOPs may not need all sections listed.

** An Operator Aid for operating equipment may just include a stepwise procedure. This aid is for an analytical procedure.

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3.0 PROCUREMENT, SUBCONTRACTING AND DOCUMENTATION

3.1 Procurement

Procurement planning begins with the PM evaluating the needs of the project including the specifications of the required items. These needs are then compared to the approved sources/vendors.

Procurement will then be done through a qualified and established vendor. When a new vendor must be used prior to qualification, the vendor must provide and/or meet any requested technical and operational specifications that may influence quality, safety, and/or environmental concerns. These specifications are reviewed with the QAO and will become part of the final project documentation. The Controller at the direction of the QAO, maintains a list of approved vendors (in the MCLinc purchasing software database).

Using a MCLinc Purchase Order, the PM documents the desired product that meets the project or use required specifications by catalog or identification number. In cases where it is necessary to assure the quality of the product, the specifications required are defined in the purchase order. The purchase order is used to confirm the material or service upon receipt.

Documentation for project related purchases are maintained in the project files and the PM is responsible to assure the specifications of products received meet project needs.

Upon receipt the procured items, they are checked by the PM or his designee, against the ordered item for compliance prior to use. The desired quality will be checked during initial use for critical consumables, supplies and services that affect the quality of MCLinc services. Any identified quality issue must be immediately reported to the QAO for investigation of root cause and determination of corrective action (See Section 2.7, Material and Sample Receipt).

For the purposes of NQA-1 requirements, MCLinc does not purchase materials for direct Nuclear Facility-Related use. All day-to-day procurements are via purchase order and are commercial grade items with specifications clearly defined by the vendor. If a unique

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item is required, the PM under the direction of the QAO/TD reviews the design and/or specifications and seeks and evaluates qualified vendors or sources. Any new vendor must be approved by the QAO.

Further details of the procurement process are defined in "Procurement Control," MCL-7727.

3.2 Subcontracting

When MCLinc uses a subcontractor for support services or testing services that it does not provide, or for workload overflow; the client is informed of this approach and a competent pre-approved subcontractor is used. The subcontractor must meet any certifications required by the project, (i.e., AIHA).

The need for subcontract services is identified in the project planning stage and if the services required are not available through a previous approved subcontractor a new subcontractor will be sought. This involves definition of the requirements for the services needed, along with any certifications required and solicitation of the supporting documentation from potential vendors. The PM will review the documentation and make a recommendation to the QAO/TD for final approval.

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4.0 NON-CONFORMANCES, CORRECTIVE AND PREVENTATIVE ACTIONS

During the course of normal business activities, problems may arise that potentially impact the quality of the work and/or MCLinc's ability to meet our client's requirements. These problems must be reported by the individual identifying the problem in a timely manner to responsible staff (QAO, TD, LM, or MCLinc President).

The problem will then be investigated and appropriate corrective action taken to resolve and eliminate future reoccurrence as required by 10CFR21, "Reporting of Defects and Non-compliance." The goal of each investigation is to determine where possible the root cause or real source of the error or variance. When found this "root cause" must be documented and become part of the lessons learned information passed on to management and staff. The QAO will randomly assess the documentation and implementation of corrective actions on quality related issues. Consideration will be given during the investigation to any preventive actions necessary to avoid future issues (e.g., change SOP or perform process spot check, etc)."

The MCLinc quality program encourages each staff member to be proactive and point out potential problem areas. Management will implement this preventive action with the same priority as any corrective action.

The non-conformance process and Problem/Action Report format is detailed in "Procedure for Reporting Problems, Non-Conformances and Associated Actions, MCL-7722."

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5.0 PERSONNEL TRAINING AND QUALIFICATIONS

MCLinc personnel are qualified to perform their job duties based upon a combination of one or more of the following criteria:

- Formal education
- On-the-job training
- Formal training (vendor courses, site and customer-specific training, etc.)

Training is performance based and proof of successful completion and understanding of the material must be demonstrated and documented. The training and qualification needs of the individual MCLinc staff members are determined by either the TD, or LM. For any new procedure the LM and TD will establish training requirements and have the analyst perform a Demonstration of Capability (DOC). This DOC procedure is explained on a DOC form available from the QAO.

5.1 Training

Personnel shall be in compliance with required training. Formal training classes will be used for the majority of the baseline and/or job-specific required training. MCLinc staff/safety meetings will be used to supplement education in safety- and technical-related issues. Off-site training (vendor schools, short courses, seminars, and conferences) can be used for continuing technical and professional training.

Training for procedure or guideline based methods is performed using required reading assignments and topical review in follow-up staff meetings. On-the-job training can be used to supplement any job performance activity. Details of MCLinc's Training Program are located in "MCL Training Program," MCL-7778.

Training records will be maintained for active laboratory personnel. The current training records for each member of the MCLinc staff will be maintained by the DCC.

New employees receive training in the following areas:

- QA/QC
- Chemical Hygiene Plan

- Health and Safety
- Radiochemistry
- Security

The level of this training will be dependent on employee job assignments, but all training will be documented and placed in their training files.

On-going updated training will be provided to all employees as required. Employees are required to read all SOPs issued to them and ask the QAO any questions or clarifications. On-going SOP training will be provided when significant changes are made to the SOP. The MCLinc staff is encouraged to suggest any training needs they have to better perform their jobs. Also, each year during the annual quality assessment, the need for additional training will be reviewed and the effectiveness of current training evaluated.

5.2 Certification of Qualification

In addition to specific training requirements, there are several areas of MCLinc operations that require special/specific qualifications. These are outlined below. If required by the project, this qualification must be further documented and clearly identify the area of qualification and the basis including, as required, any supporting documentation. See example Certificate of Qualification in Appendix E.

5.3 Instrument Operator Qualifications

Operator status will be determined and confirmed by either the TD or the LM. Appendix D lists the instrumentation which requires approval and the MCLinc personnel that are authorized (as of the date shown) as operators. The QAO is responsible for maintaining and distributing updates for the authorized operator listing.

5.4 Radiological Materials

MCLinc staff members must meet the training and authorization requirements as defined by the RSO. The specific requirements for radiological use authorization are defined in the Tennessee Department of Environment and Conservation, Division of Radiological Health, Application for Radioactive Material License and/or the DOE Radiological Protection Program.

5.5 Classified Materials

MCLinc staff members must meet all of the requirements specified in the "Facility Security Plan," MCL-7706. The most important criteria is the need-to-know. This aspect of control over the unauthorized distribution of classified and controlled information will be fully enforced within all aspects of MCLinc business practice. The FSO will maintain all associated documentation and records that are required for compliance with the Facility Security Plan.

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6.0 TECHNICAL PROGRAMS

Since the vast majority of MCLinc's technical programs are non-routine or first time research driven activities, the work is performed based on the work plan or scope of work agreed upon with the client. The guidance to execution of their work is found in the MCLinc SOPs including Project Management and Instrument Operational Guides. The MCLinc President and the QAO must approve MCLinc SOPs. All laboratory work is documented in laboratory notebooks to assure recreation of the process followed. The other various guidelines, procedures, and plans that form the basis for the operations and quality performance of MCLinc are listed in the Reference Section of MCLinc's Controlled Documents, Volumes I, II and III.

6.1 Pre-Project Activities

Consideration will be given to the quality, safety and environmental impacts on project performance during the project conception, bidding, procurement, and initiation phases. These areas will be addressed either informally or formally for all MCLinc work. These issues will be documented when dealing with a customer whose work scope is estimated to take more than 80 man-hours to complete. This consideration will help ensure that all customer and MCLinc data quality objectives can be established and met during the successful completion of the work scope.

6.2 Project Conception

Project ideas will be reviewed by MCLinc staff members to determine if the work being requested or proposed is within the capabilities of the MCLinc staff, facility, and resource allocation. Consideration as to resources, facility requirements, waste generation/disposal, and total project life cycle costs and requirements will be considered. Discrepancies or concerns will be presented to either the TD or LM to obtain resolution on the discrepancies or concerns.

6.3 Bidding

When providing a cost estimate or quote for a specific set of services to a customer, the PM will have the cost estimate reviewed by either the MCLinc President, TD, or LM. The internal cost breakdown analysis will demonstrate that consideration has been

provided to meet the customer's quality, documentation, reporting, sample management, procurement, and waste disposal requirements within the cost estimate being provided.

6.4 Project Acceptance

Before a project is accepted and begun, the PM will meet with the LM and TD to make sure that all required MCLinc resources will be available and can be allocated for the successful completion of the customer work package. This includes a review of any possible procurement of goods and services to complete the project.

6.5 Project Documentation and Communication

At the discretion of the PM and the customer, the amount of project specific QA documentation and procedures will be determined. These documents are used as guidance and can be informally approved and accepted between the PM and the customer. The documents will be part of the permanent project file and are the responsibility of the PM to ensure that all proper documentation is archived. The project specific documentation may include but is not limited to:

- QAP,
- Data package/reporting requirements,
- Specific technical procedures or operational methods,
- Enhanced Chain of Custody procedures,
- Calibration and/or certification requirements,
- Environment, health and safety (EH&S) guidance,
- Budget, schedule, and deadline information, and/or
- Correspondence.

A key element of the MCLinc Project is effective communication to the client not only of the project problems or issues, but progress and significant achievements. This communication also allows an opportunity for input to the project from the client on technical matters, opinions and interpretations of the results. In most cases this input is best received during the project than after the fact. The mode of this communication is best dictated by the client and may mean phone calls, meetings, e-mail or other written progress reports. Document all oral communication to assure your understanding of the discussion.

6.6 Reporting and Project Closure

Report structure, detail, organization, and media selection will be determined by the PM and customer. The PM will ensure that all data reviews, data tabulations, laboratory work, and customer requests have been fully completed and documented prior to the compilation of the final report. Any non-conformity with the customer's request will be communicated and documented as soon as possible with the customer. Documented resolution will be noted within the project notes and summary. The PM will ensure that a complete data set,

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laboratory notebook reference list, data location listing, and final copy of the customer report are maintained in their files. A copy of the final customer report will be maintained within the MCLinc project files. If a report is to be amended, the report is either reissued or marked as a new revised version, or a clearly defined amendment to the report is issued. In both cases, the new document is sent to the same distribution as the original report.

Upon completion of the project, the PM must place or reference all applicable documents in the project file, make sure any non-routine or special wastes generated during the project are properly stored and/or disposed per "Waste Management Plan," MCL-7718, and that all samples and residues are properly stored for disposal or returned to the client per the contract.

6.7 Clients Complaints

MCLinc welcomes feedback from our clients, be it positive or negative. Despite the efforts to the contrary, the probability exists that the client may express concerns or disfavor with the project to MCLinc staff. Anyone aware of such a complaint must report it to the appropriate QAO, PM or TD for follow-up. It is critical that MCLinc understand completely both sides of the complaint, the root cause and take immediate corrective action. The complaint will be documented with a nonconformance or corrective action memo per MCL-7722, "Procedure for Reporting Preventive Actions, Problems, Non-conformances, and Associated Actions." The QAO will review this corrective action and discuss with client as necessary. Since the majority of our projects include direct contact with the client, discussions concerning their satisfaction or dissatisfaction with our work can be held one on one with any staff member.

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7. 0 DOCUMENT CONTROL PROCEDURES

7.1 Document Control

Documents will be managed to ensure that a consistent record of activities exists to allow for a detailed review of current practices to determine if any modifications would permit the improvement of any process, in part or in whole. Documents which are determined to be important to the operation and control of materials and information within MCLinc will be controlled. Controlled documents will be maintained with respect to "Document Control," MCL-7703. Examples of controlled documents are:

- QAPs
- Quality Documents prepared for clients
- Standard Operating Procedures
- Chemical Hygiene Plans
- Health and Safety Plans
- Waste Management Plans
- Operator Aids

During the annual internal audit by the QAO, all controlled documents will be reviewed and if revisions are necessary, they will be scheduled and implemented. Those not revised will be marked "Reviewed without Revision with the date" in the Controlled Document Status form.

7.2 Changes to Controlled Documents

Changes to controlled documents may be initiated by anyone using the document to clarify or correct an error or reflect a change in the procedure. Changes shall be reviewed and approved by the same functions that approved the original document. Information needed to evaluate the requested change, if necessary, should be provided along with the MCLinc Change Form (Example in SOP, "Document Control," MCL-7703). The changes shall be noted on the change form and if required for clarity attachment of the revised document pages. Once signature approval is complete the DCC will issue a controlled copy of the change form and any attachments to all recipients of the original controlled document.

7.3 Notebooks

A critical document in use within MCLinc to record day-to-day work efforts, analyses, and experimentation is the laboratory notebook. Laboratory notebooks are issued by the DCC to individual personnel. These notebooks are assigned with a unique identification number and are maintained by the individual MCLinc personnel. The notebook is the responsibility of the individual user. It is good practice to maintain an index in the front of the book to track the time frames associated with various customers and/or projects which have documentation in the notebook. If it is felt that a section or entry into the logbook should be witnessed, the logbook owner is responsible for providing another cognizant MCLinc staff member to read, verify, and sign the logbook pages that the material has been properly documented and dated. Notebooks, when completed or retired, are returned to the DCC for safe storage. Notebook(s) will be randomly reviewed for compliance to the SOP, "Good Notebook Keeping Practices," MCL-7724, during the year as part of each internal assessment by QA or the TD.

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8.0 RECORDS

8.1 QA Records

Records that show or demonstrate evidence of quality or a quality system are deemed quality records. They are to be legible, identifiable and retrievable. QA records may be hard copy or electronic media files. Quality records are maintained by the DCC and the QAO and include the following:

- Current and historical controlled documents
- Laboratory notebooks
- Laboratory / instrument logbooks
- Training files/records
- Instrument output, results, notes, design documents and calculations
- Standards traceability documentation
- Radiochemical inventory documentation (maintained by RSO)
- Non-conformance reports
- Demonstration of Capability Form (DOC)

The PM maintains QA Records that are specific to a project such as standard runs, daily calibrations, calculations and results in the project files.

8.2 Project Records

All technical and business records associated with a project constitute Project Records and are maintained accessible to the project staff during the project and are considered client proprietary. The technical records are maintained by the PM in the designated project files and the business records by the DCC in the Administrative Office files. Examples of Project Records are:

- Work plans or scope of work documents
- Project QAPs
- Project correspondence including phone logs
- Interim and final reports
- Computer files of project information
- Proposals, contracts and change orders

Further details on records are outlined in "Quality Assurance Records,"MCL-7729.

8.3 Record Retention

Record retention is the key to assuring our clients that information if needed in the future is retrievable. Project records are maintained for five (5) years or as otherwise defined in the project contract. QA Records not associated with a project are considered lifetime or permanent records and will be maintained for the usable life of the item. All records are maintained within the secure MCLinc facility in clean, dry areas with access controlled by the The Sample/Report Management Staff (SRM). The SRM receives project documents from the MCLinc staff and places the records into appropriate filing cabinets or new storage boxes and logs the contents into a records storage log which is then used to track the documents for future retrieval. All documents in storage are accessible only through the SRM or QAO.

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9.0 ASSESSMENTS

9.1 Management Assessment

Ultimate responsibility for QA/QC and ES&H compliance within MCLinc rests with the MCLinc President. Unresolved MCLinc issues will be resolved by MCLinc management. MCLinc evaluates its performance in January for the previous year with an Annual Management Quality Assessment in January of each year. During this Annual Management Quality Assessment, issues are raised, resolved and documented. The purpose of the Annual Management Quality Assessment is to provide a means to understand the effectiveness of the management system, make recommendations for improvement to top management and implement the improvements. Tools like the quality policy, client and laboratory QA objectives, performance test (PT) sample results and internal and external assessments are used to allow these improvements while maintaining the integrity of the system.

As part of the MCLinc Annual Management Quality Assessment, the MCLinc management review shall take account of:

- Quality objectives of management met
- The suitability of policies and procedures
- Reports from management/supervisory staff
- Results of internal or third party audits/assessments
- Corrective and preventive actions
- The results of round robin or any PT program
- Changes in volume and type of work
- Client feedback or complaints
- Manpower/equipment needs
- Staff recommendations for improvement
- Other relevant factors such as QC activities resources and staff training

Upon completion of the draft Annual Management Quality Assurance Review, the document is submitted to the MCLinc President/Chief Executive Officer (CEO) for review and determination of any findings. Any findings resulting from this management

review will be defined with a designated person responsible and an agreed upon time schedule.

The management assessment will be documented by the QAO.

9.2 Internal Assessments

The QAO on an annual basis will schedule and initiate assessments of the internal quality systems of all the laboratory operations using a total review or checklist approach and documenting all findings in a memo report. These assessments may be performed in part periodically (i.e., monthly) or on a single event. The QAO will define or approve the corrective actions and follow-up as necessary to assure corrective actions have been implemented. A third party independent quality systems assessment sponsored by MCLinc meets the requirement for this assessment. Appendix F is the 2014 MCLinc assessment schedule.

9.3 Independent Assessments

Clients or MCLinc may utilize other organizations, independent of the day-to-day operations of the MCLinc facility, to provide an assessment of quality, safety, and environmental activities within the MCLinc facility. MCLinc will provide a safety orientation to the members of the independent assessment team at the beginning of the assessment kick-off meeting.

All documentation generated by the independent assessment will be addressed in a closeout report that will be generated by the appropriate MCLinc staff no later than twenty-five (25) working days after the independent assessment results are presented to management. Corrective actions will be documented and their effect on the deficiency tracked and noted. If it is felt that the corrective action has had a significant impact on other areas of operation, the corrective action documentation will be used by the appropriate MCLinc staff to compile a positive lesson learned document to ensure that all portions of the MCLinc organization is aware of the potential positive influence of the corrective action. MCLinc may initiate a third party additional audit for specific areas of the laboratory or total laboratory operation.

9.4 Performance Evaluation (PE) and Performance Testing (PT)

MCLinc will participate in PE and PT programs as necessary to evaluate the quality performance of the laboratory. MCLinc currently participates in Mixed Analyte Performance Evaluation Program (MAPEP) (Inorganic and Rad - soil and water); AIHA for metals, air asbestos, beryllium oxide, and bulk asbestos; internal PEs for hexavalent chromium and mercury; and a third-party asbestos program. Others will be added as needed. Any non-passing score in these programs will be investigated and a written report submitted to the QAO within 21 calendar days. *Supplemental PE samples are hex-chrome in water and PCBs in oil, both bi-annual.* The QAO will approve and follow-up on the corrective actions as needed.

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10.0 REFERENCES

ANSI/ASQC E4-1994, "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs."

ASME NQA-1-2000, Edition, "Quality Assurance Program Requirements for Nuclear Facilities."

DOE, "Consolidated Audit Program Quality Systems for Analytical Services Revision 2.9."

Energy, Nuclear Safety Management, Quality Assurance Requirements, Scope, 10 CFR Part 830.120.

Energy Reporting Defects and Noncompliance, 10 CFR Part 21.

ISO/IEC Standard 17025 – "General Requirements for the Competence of Testing and Calibration Laboratories, 2005."

MCL-7702, "Chemical Hygiene Plan."

MCL-7703, "Document Control."

MCL-7704, "Project Management Guide."

MCL-7705, "Nuclear Materials Control and Accountability Plan."

MCL-7706, "Facility Security Plan."

MCL-7708, "Electron Microscopy Operation Guide."

MCL-7710, "Sample Preparation Guide."

MCL-7711, "Calibration, Inspection, and Maintenance Guide."

MCL-7715, "Radiation Protection Plan."

MCL-7717, "Health and Safety Plan."

MCL-7718, "Waste Management Plan."

MCL-7719, "Asbestos Laboratory Quality Assurance Manual."

MCL-7722, "Procedure for Reporting Problems, Non-Conformances, and Associated Actions."

MCL-7724, "Good Notebook Keeping Practices."

MCL-7727, "Procurement Control."

MCL-7728, "Verification of Data Software."

MCL-7729, "Quality Assurance Records."

MCL-7735, "Estimation of Uncertainty of Measurement (EUM)."

MCL-7756, "Operator Aids."

MCLinc's Controlled Documents, Volumes, I, II, and III.
"National Environmental Laboratory Accreditation Conference Standards," Latest Approved Edition

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MCLinc President	Date 9/1/15	Section: Appendices Section No: 11 Revision: 14 Date: 09/01/2015
Quality Assurance Officer	Date 9/1/15	

Appendix A - MCLinc QAP Cross-reference to National and International Quality Requirements

Basic Requirements of NQA-1	Requirements of ISO/IEC 17025	MCLinc QAP Section	10 CFR 830.120	ANSI/ASQC E4-1994
Organization	Organization and Management	1.0 Organization	Management	Management and Organization
Quality Assurance Program	Management System Personnel	2.0 Management Systems 5.0 Personnel Training and Qualifications	Quality Assurance Program; Personnel Training and Qualifications	Quality Systems and Description; Personnel Qualifications and Training
Design Control		2.13 Data Review and Evaluation	Design	Design
Procurement Document Control	Review of Requests, Tenders and Contracts	3.0 Procurement, Subcontracting and Documentation	Procurement	Procurement; Planning and Scoping
Instructions, Procedures, and Drawings	Technical Requirements	6.0 Technical Programs	Documents and Records	Documents and Records; Design of Systems
Document Control	Document Control	7.0 Document Control	Documents and Records	Documents and Records
Control of Purchased Items and Services	Purchasing Services and Supplies	3.1 Procurement	Procurement	Procurement
Identification and Control of Items	Measurement Traceability	2.1 Standards and Reference Materials	Procurement	Procurement
Control of Processes	Accommodation and Environmental Conditions	2.0 Quality Systems	Performance-Work Processes	Implement Work Processes; Operation of Systems
Inspection	Subcontracting of Tests and Calibrations	2.12 Data Review 3.2 Subcontracting 9.2 Internal Assessments	Inspection and Acceptance Testing	Computer Hardware/Software; Implementation of Planned Operations
Test Control	Assuring the Quality of Test and Calibration Results	2.2 Calibration	Quality Improvement	Quality Improvement
Control of Measuring and Test Equipment	Test and Calibrations Methods and Method Validation Equipment	2.2 Calibration 2.11 Instrumentation	Quality Assurance Criteria	Quality Systems
------	Service to the Client; Complaints	6.0 Technical Programs	Quality Assurance Criteria	Planning and Scoping
Handling, Storage, and Shipping	Sampling	2.10 Sampling and Sample Preparation	------	Design of Data Collection
Inspection, Test and Operating Status	Handling of Test and Calibration Items	2.0 Quality Systems	Inspection and Acceptance Testing	Design of Data Collection/Verification and Acceptance
Control of Non-Conforming Items	Control of Non-conforming Testing and/or Calibration Work	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Corrective Action	Corrective Action Preventative Action	4.0 Non-conformances, Corrective and Preventative Actions	Quality Improvement	Quality Improvement
Quality Assurance Records	Reporting of Results Control of Records	6.6 Reporting 8.0 Records	Documents and Records	Documents and Records
Audits	Internal Audits Management Reviews	9.1 Management Assessment 9.2 Internal Assessments 9.3 Independent Assessments	Management Assessment; Independent Assessment	Assessment and Response

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Appendix A Cross Reference (Continued)

DOE Quality Systems for Analytical Services Latest Revision		MCLinc Quality Assurance Plan Revision 14	
Section	**Title**	**Section**	**Title**
1.0	Introduction, Scope, Applicability	1.1	Introduction
2.0	References	10.0	References
3.0	Terms and Definitions		Defined throughout QAP
4.0	Management Requirements	1.0 2.0	Organization Management Systems
4.1	Organization	1.0	Organization
4.2	Management	2.0	Management Systems
4.3	Document Control	7.1	Document Control
4.4	Review of Requests, Tenders and Contracts	6.3	Bidding
4.5	Subcontracting of Environmental Tests	3.2	Subcontracting
4.6	Purchasing Services and Supplies	3.1	Procurement
4.7	Service to the Client	6.5	Project Documentation and Communication
4.8	Complaints	6.7	Client Complaints
4.9	Control of Nonconforming Environmental Testing Work	4.0	Non-Conformances and Correction Preventative Action
4.10 4.11	Improvement Corrective Action	4.0	Non-Conformances and Correction Preventative Action
4.12	Preventive Action	4.0	Non-Conformances and Correction Preventative Action
4.13	Control of Records	8.0	Records
4.14	Internal Audits	9.2	Internal Assessments
4.16 4.17	Management Reviews Data Integrity Investigations	9.1	Management Assessments
5.0	Technical Requirements	2.0	Management Systems
5.1	General	2.0	Management Systems
5.2	Personnel	5.0	Personnel Training and Qualification
5.3	Accommodation and Environmental Conditions	2.4	Work Environment
5.4	Environmental Test Methods and Method Evaluation	2.14	Standard Operating Procedures
5.5	Calibration Requirements	2.11	Instrumentation and Maintenance
5.6	Measurement Traceability	2.1 2.5	Standards and Reference Materials Laboratory Supplies
5.7-5.8	Sampling and Handling of Samples	2.7 2.8 2.10	Material and Sample Receipt Controlled Samples Sampling and Sample Preparation
5.9	Quality of Environmental Test	2.12 2.13	Quality Control Samples and Assessments of Data Data Review and Evaluation
5.10	Reporting the Results	6.6	Reporting and Project Closure
6.0	Hazardous and Radioactive Materials Management and Health and Safety Practices		Various Locations and Two Separate SOPs (MCL-7718;& MCL-7717; and MCL-7715)
6.1	Radioactive Materials Management and Control	5.4	Radiological Materials and Radiation Protection Plan, MCL 7715 SOP
6.2	TSCA [Toxic Substance Control Act of 1976] Materials		Chemical Hygiene Plan MCL 7702 SOP
6.3	Laboratory Health and Safety		Health and Safety Plan MCL 7717 SOP
6.4	Waste Management and Disposal		Waste Management Plan MCL 7718 SOP

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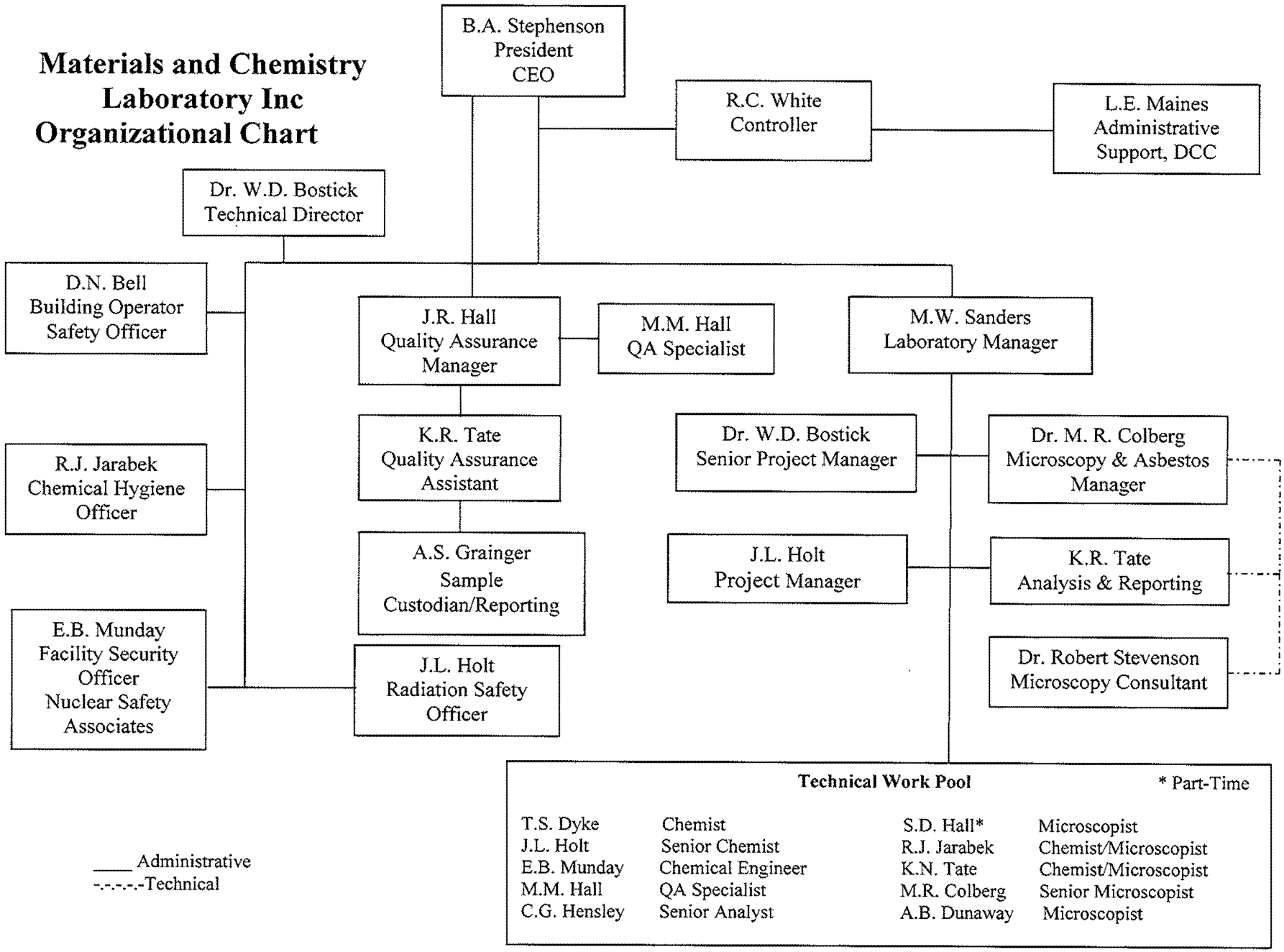
Materials and Chemistry Laboratory Inc Organizational Chart



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Appendix C - MCL Support Function Assignments

Position	Personnel
Company Controller	Robert C. White
Chemical Hygiene Officer	Robert J. Jarabek
Classified AIS Security Site Manager	Mark R. Colberg
Classified AIS System Security Officer	Earl B. Munday with Charlie Coffey
Classified Document Custodian	Earl B. Munday
Classified Document Custodian - Alternate	Robert J. Jarabek
Document Control Coordinator	Linda E. Maines
Facility Security Officer	Earl B. Munday
Facility Security Officer - Support	*Atkins Nuclear Solutions US*
MBA Custodian(s)	Robert J. Jarabek Earl B. Munday
NMC&A Manager	Earl B. Munday
NMC&A Alternate Manager	*Mary M. Hall*
OPSEC Manager	Phyllis Ferguson
OPSEC Alternate Manager	Earl B. Munday
MCLinc President	Barry A. Stephenson
Laboratory Manager	Michele W. Sanders
QA Officer	Jack R. Hall
QA Officer-Alternate	Mary M. Hall
Radiological Safety Officer	Jeff L. Holt
Radiological Safety Officer - Alternate	Michele Sanders
Security Container #1 Custodian	Earl B. Munday
Security Container #1 Custodian - Alternate	Mark R. Colberg
Security Container #2 Custodian	Earl B. Munday
Security Container #2 Custodian - Alternate	Robert J. Jarabek
Security Container #3 Custodian	Mark R. Colberg
Security Container #3 Custodian - Alternate	Earl B. Munday
Safety Officer	David N. Bell
Technical Director	William D. Bostick

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Appendix D – Instrumentation with Responsible Owner and Authorized Operator

Type	Model	Manufacturer	MCLinc Owner
FTIR	MB100	BOMEN	Munday
FTIR	GL3020/Nicolet (6400)	Mattson	Bostick/Tate
GC (3) 2EC + FID	5890 (2), 7890A	Hewlett Packard	Sanders/ Holt/Tate
IC	ICS1100	Dionex	Sanders/Holt/Dyke/Hensley
ICP	2000	Perkin Elmer	Sanders/Jarabek/Holt/Dyke
ICP/MS	Elan 9000	Perkin Elmer	Sanders/Holt
Mercury Analyzer (AA Cold Vapor)	410	Buck	Sanders/Dyke/Hensley
Mercury Analyzer (Low Level)	Hydro C	Teledyne-Lehman	Holt/Hensley*
Mercury Analyzer (Low Level)	Hydro II AF GOLD	Teledyne-Lehman	Holt/Hensley*
Optical Microscope	Various	Various	Colberg/Jarabek/D. Hall/ Tate/Holt
Rad Spectroscopy	Various	Various	Jarabek/Bostick
SEM	840	JEOL	Colberg/Dunaway*
SEM	ESEM-2020	Philips	Colberg/Dunaway*
SEM	S-4500	Hitachi	Colberg/Dunaway*
SEM	S-5000	Hitachi	Colberg/Dunaway*
TEM	2000FX	JEOL	Colberg/Dunaway*
UV-Vis Spectroscopy	PC1000	Ocean Optics	Bostick
XRD	MiniFlex II	Rigaku	Colberg/Tate
TGA/DTA+MS+GC	6300 ThermoStar GSD301/8610C	SII/Pfeiffer/SRI	Tate/Sanders

*In training

AA-Atomic Absorption
DTA – Date Transfer Analyzer
EC – Electron Capture
FID – Flame Ionization Detector
FTIR – Fourier Transform Infrared Spectroscopy
GC – Gas Chromatography
IC – Ion Chromatography
ICP – Inductively Coupled Plasma
MS – Mass Spectroscopy
SEM – Scanning Electron Microscope
TEM – Transmission Electron Microscope
TGA – Thermogravametric Analysis
UV-Vis – Ultraviolet – Visible
XRD – X-Ray Defraction

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Appendix E - CERTIFICATE OF QUALIFICATION AND AUTHORIZATION

MCLinc CERTIFICATE OF QUALIFICATION

Certification of: _____

Certified To Perform: _____

_____

_____

Certification based on:

- ☐ Education
- ☐ Indoctrination
- ☐ Experience
- ☐ Training
- ☐ Test Results (Attach)
- ☐ Capability Demonstration:

  (Observed by: _____)

Certification Level (I, II, III, per NQA-1): _____

Technical Director/QAO Approval: _____

Date of Certification: _____

Expiration Date: _____

Results of Periodic Evaluation: _____

_____

_____

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Appendix F

MEMO

DATE: December 30, 2014

TO: Barry A. Stephenson, MCLinc Staff

FROM: Jack R. Hall

FAX#: (865) 576-8558 PHONE: (865) 574-9923

SUBJECT: McLinc QA Internal Assessment Schedule for 2015

MEMO:

As part of the MCLinc QA Plan there is a requirement for an annual assessment, which I am scheduling to occur over the year covering the various quality systems. The schedule is as follows:

January/February	Complete 2014 Management Assessment /Radiation Plan Review-Assessment
March/April	Project files, QA files, and Training files
May/June	Review QAP/ Corrective/Preventative Action Process
July/August	Instrument/Equipment Calibration/Reference Materials + Update QAP
September/October	Sample Log-in Process/ Random Notebook Review
November	Industrial Hygiene Laboratory Internal Assessment per AIHA
December	Procurement and Document Control and perform QA SOPs Review

As part of these quality systems assessments I will be asking for your help to take any needed corrective actions.

Materials and Chemistry Laboratory, Inc.
East Tennessee Technology Park, Building K-1006
2010 Highway 58, Suite 1000, Oak Ridge, Tennessee 37830-1702
Phone: (865) 576-4138 Fax: (865) 576-8558

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MATERIALS & CHEMISTRY LABORATORY, INC. (MCL)
STANDARD OPERATING PROCEDURES

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INFORMATIONAL USE ONLY

MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE		
Operation Guidance Electron Microscopy: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President	2/17/2009 Date
	Quality Assurance Officer	2/16/09 Date

1.0 INTRODUCTION

This document and the documents referenced herein provide a framework for the safe and consistent operation of electron microscopes. It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage along with providing a high level of confidence in the results obtained.

2.0 GENERAL RESPONSIBILITIES

2.1 Principle Operator

The Principle Operator is responsible for the routine operation, upkeep of the instrumentation, documentation, and work area associated with the instrumentation. The appointment of the Principle Operator for each instrument is made by the *Chief Operating Officer.*

2.2 Secondary Operator

The Secondary Operator should be able to assist the Primary Operator in routine operation and maintenance. The Secondary Operator may be able to perform all operations at the same level of expertise as the Primary Operator, but this is not a requirement. Secondary Operators may be certified by either the *Chief Operating Officer* or the Principle Operator.

2.3 *Chief Operating Officer*

The *Chief Operating Officer* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1 Major Components

This table lists the major equipment covered by this guideline. The property number is the property number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #
Hitachi	S-4500	K333391	A104
JEOL	JXA-840	K322408	A106
JEOL	2000FX	K331267	E101
Hitachi	S-5000	K333393	E102
FEI	ESEM-2020	K333395	E103

3.2 Basic Process Description

Electron microscopy (EM) impinges a focused electron beam on a solid surface to produce electron *images* and x-rays which contain information about the sample. The electrons are used to create electron micrographs (images) and the x-rays are used to obtain elemental information about the sample *with associated x-ray analyzers*. EMs vary by the nature and relative position of their electron optic components with respect to the sample. EMs can optimize various electron-sample interactions (i.e. scanning, transmission, and diffraction) to obtain various types of materials characterization. The "output" is typically an electron micrograph *from a secondary electron detector (SEI), backscattered electron detector (BEI), or transmitted onto a fluorescent screen*, electron diffractogram, or elemental *composition by x-ray spectroscopy* (qualitative or quantitative). The following are brief overviews of typical operational aspects of the instrumentation:

> The electron guns operate at very high voltage (1,000 to 200,000 volts) but at very low current (nA to pA range). EMs operate in a vacuum with the electron gun typically being at 10^{-6} to 10^{-7} torr and the sample being between 50 and 10^{-5} torr; hence, sample exchange and manipulation are done via sample exchange interlocks and mechanical stages.
>
> *Each instrument has associated equipment required for the vacuum system, cooling, and valving (compressors). Operators are required to understand the interaction of each component and perform routine, preventive maintenance on each component according to the vendor operating manual.*
>
> *Each scope has an associated x-ray analyzer used to determine elemental composition. X-ray emission is shielded by the metal construction of each instrument.*

3.3 *Basic Operating Process*

This describes the *general* guidelines for *sample preparation, instrument operation, and collecting & transferring data for interpretation.*

3.3.1 *Sample Preparation*

Sample preparation for SEM and TEM investigation is the key for a successful investigation. The following notes should be considered prior to loading a sample into the electron scopes:

SEM: Loose powders are not acceptable in the SEMs. SEM preparations are typically mounted on adhesive carbon tape on top of graphite planchets (ie. Ted Pella, Inc.). Because of sample charging, samples are typically carbon coated to reduce the effect of charging on the images. Feature mapping under a stereoscope prior to analysis is strongly recommended to help navigate on the sample at the higher SEM magnifications.

TEM: Loose powders are not acceptable in the TEM. TEM copper grids with a Formvar film layer can be purchased from Ted Pella, Inc. Three microliter samples can be mounted directly on these grids, dried, and loaded into the TEM. A dispersion in ethanol with gentle sonication works well. The technique for preparing a TEM grid for NIOSH 7402 is outlined in the NIOSH 7402 procedure and MCLinc SOP 7742.

3.3.2 Instrument Operation

Each instrument has a unique start-up/shutdown procedure outlined in each vendor manual. Instruments must be operated according to the vendor operating manual which outlines procedures for loading/unloading samples, operation, data collection, maintenance, and troubleshooting.

Note that the EMs should never be left unattended when the electron source is activated. When not in use, the EMs should be left in the shutdown condition outlined by the principle operator.

3.3.3 SEM Data Collection and Transfer

The SEMs can collect electron images from 20x to 1,000,000x magnification. Both SEI and BEI images can be collected and stored. Images can be transferred for reporting by:

- Polaroid film: Each SEM unit has been set up to collect images by Polaroid type 52 or 57 land film. Film development takes less than 1 minute and has excellent resolution.

- Printer: Each associated x-ray analyzer has the capability to grab the image from the SEM's CRT and print to a printer. The image can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving images in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

X-ray spectra can be transferred for reporting by:

- Printer: Each associated x-ray analyzer has the capability to print to a printer. The spectra can then be scanned and converted to a electronic data file.

- Electronic Data File: The Hitachi 4500 has an EDAX x-ray analyzer that is capable of storing and saving spectra in various formats including bmp, tif, and jpg formats. Data is readily transferred by memory card or CD.

3.3.4 TEM Data Collection and Transfer

The JEOL 2000FX TEM can collect electron images from 20x to 1,000,000x magnification. Images can be collected and stored only by Kodak film. Follow manufacturer's instructions for using Kodak D-19 Developer and Kodak Rapid Fixer.

X-ray spectra can be transferred for reporting by a printer associated with the x-ray analyzer. The spectra can then be scanned and converted to a electronic data file.

3.4 Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

Disposable lint-free or powder-free gloves
Lint-free cloths
Disposable laboratory waste bags
Fume hoods equipped to provide a well-ventilated workspace
Protective eyewear
Protective laboratory coat/apron
Spill cleanup material
Emergency eyewash station
Emergency shower station
Fire extinguisher
Access to MSDS sheets for all chemicals used

Ted Pella, Inc and SPI, Inc. are good sources for various EM supplies for sample preparation such as TEM grids and graphite planchets.

3.5 Standards

The following components, or equivalent ones, should be available for quality control and performance evaluation of the various electron microscopes. The selection and use of the particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook and should be used in agreement with the methods outlined in this document.

Magnification standards:

- NIST 484A Specimen ID JY-55-OJ (2 each)
- NIST 484E Specimen ID-SH
- 2160 lines per millimeter cross grating (E. F. Fullam, Inc., Cat. #60021)

Elemental standards:

- C. M. Taylor Corp. #1 Element STD 202-52
- C. M. Taylor Corp. #2 Element STD 202-52
- C. M. Taylor Corp. #4 Element STD 230-27
- C. M. Taylor Corp. #5 Element STD 230-30
- SPI STD 87-103
- Tousimis 8026 103-S

X-ray performance (FWHM) standards:

- X-checker, Small World (#1)
- X-checker, Small World (#2)
- C. M. Taylor Corp. #1 Element STD 202-52

Resolution standards:

- Prickly gold grid Type D

These standards are centrally located, in dry boxes where applicable. Control is maintained through storage in manufacturers labeled containers or in labeled sample storage containers. The standard certification papers are filed with the QA Officer.

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

Follow guidance outlined in the Chemical Hygiene Plan for the Materials and Chemistry

Laboratory, Inc. (MCL-7702) and the Quality Assurance Plan to the Materials and Chemistry Laboratory, Inc. (MCL-7701).

Develop and encourage safe laboratory habits.
Food will not be stored or consumed in lab areas.
All work areas are to be kept clean and uncluttered.
Safety glasses are required to be worn as posted.
The appropriate personal protective equipment must be worn when required by the job.
Report accidents and near-miss accidents to your supervisor.
On-the-job injuries must be reported immediately.

4.2 Specific Hazards

The electron guns operate at very high voltage (1,000 to 200,000 volts). When changing a filament or performing maintenance, the vendor operating procedure must be followed exactly to prevent high voltage exposure.

For specific hazards of the instruments see the operator's manual and MCL-7717 for Health and Safety approaches to handling the hazards properly. Do not operate unless you understand potential hazards involved with the instrument.

4.3 Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print, signed, dated, and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

Kodak Rapid Fixer - Used fixer will be sent out for resource recovery of silver.
Polaroid Film Packs - Digital images will minimize film waste.

Sample Preparation - Use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

Reuse sample planchets by using small amount of double sticky carbon tape. The carbon tape and sample can be peeled off after the analysis and disposed of as solid waste. The planchet can then be reused to mount samples without being added into the waste stream.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with the MCLinc Waste Management Plan MCL-7718.

5.3 Environmental Risks

Routine operation of this equipment poses no environmental risks.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The following information should be documented at a minimum of the time period stated and after maintenance activities have been performed. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. Documentation is the responsibility of the Principle Operator and will be kept with the instrument.

Image magnification (at least semiannually): A determination of the magnification of applicable image source(s) shall be performed.

Energy calibration (at least semiannually): The energy calibration shall be checked. Standards such as Cu and/or Al should be used.

EDS energy resolution - FWHM (at least semiannually): A Mn Ka peak shall be used to measure the full width at half maximum peak intensity (FWHM).

WDS performance (as needed): The position and FWHM of peaks of interest will be documented.

Performance Documentation

The following information shall be documented in the time period stated.

Instrument usage (every time): Logbooks shall be kept for each EM to record instrument usage, operator and project number.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the service provider should be kept in chronological order. Any information or work which has been provided in response to questions or operational abnormalities that is not clearly documented in the paperwork should be documented and attached.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in section 6.1 should be documented.

6.3 Vendor Manuals

Vendor manuals form the basis of documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be readily available during instrument operation.

6.4 Data Tracking

Data documentation and archival information is the responsibility of the originator and should be recorded in the laboratory notebook.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance Plan* (MCL-7701)

MCLinc *Waste Management Plan* (MCL-7718)

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MATERIALS AND CHEMISTRY LABORATORY, INC. STANDARD OPERATING PROCEDURE	
Operation Guide Sample Preparation: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President — 4/7/10 Date Quality Assurance Officer — 3/22/10 Date

1.0 PURPOSE

This document and the documents referenced herein provide a framework for safe and consistent sample preparation. It is assumed that operating personnel have a basic understanding of the sample preparation methods. This guideline will identify the hazards associated with sample preparation and ensure the safe usage of the instruments and methods along with providing a high level of confidence in the results obtained and hence provide the foundation for a quality control and quality assurance program.

It is noted that this plan does not specifically address all aspects of the Asbestos Analysis Laboratory (AAL). The AAL is an American Industrial Hygiene Association (AIHA) accredited laboratory. The AAL operates under a stand-alone quality plan, MCLinc *Industrial Hygiene Laboratory Quality Assurance Manual* (MCL-7719).

This SOP provides general guidelines to sample preparation where as specific sample preparation details may be found in the SOPs appropriate for the analysis.

2.0 ROLES

2.1 Technical Staff

The MCLinc member performing any of the various methods of sample preparation.

2.2 Operations Manager

The Operations Manager represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required equipment.

3.0 EQUIPMENT AND MATERIALS

3.1 Major Components

The use, application, calibration and maintenance of various laboratory components are documented in supplemental QA documents. The proper use and documentation of performance of any equipment used for sample preparation is the responsibility of the individual user.

3.2 Basic Process Description

Sample preparation can be the most important part of an analysis. Yet at the same time it is the most free-style activity of the analysis. Sample preparation utilizes various tools to optimize often the desired characteristic of the sample for the desired analysis. Examples of this include but are not limited to:

- Dispersion of sample for particle size analysis.
- Coating of sample to improve the charge transfer capabilities of the sample.
- Thin layering of a sample to provide bulk analysis but still obtain proper coating.
- Degrease the surface to optimize the sample surface exposure.
- Dry, grind, sieve, size separation, or other physical alterations.
- Prepare epoxy mounts for viewing and analysis or incorporate sample into epoxy mount for viewing and analysis.
- Density and/or size separation and selective sub-sampling.
- Weigh, measure or sub-sample to obtain a known representative working sample.

The many options that exist for sample preparation are often developed over years of trial and error and are best suited to the individual preference. Thus, when performing sample preparation it is important to be aware of the hazards associated with the specific chemicals that are being used as well as the instrumentation and tools being used. It is important to document the methods used for sample preparation to optimize the process (make sure that inappropriate methods are not repeated) and make sure that good methods can be repeated if necessary. Sometimes it is the exact combination of sample and method that works and hence is not reproducible for other samples. It is important that new methods of sample preparation are not performed during off-shift hours.

3.3 Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available to ensure a safe (personnel and environmental) work place.

- Disposable gloves
- Disposable laboratory waste bags
- Fume hoods equipped to provide a well-ventilated work space
- Protective eye wear
- Protective laboratory coat/apron
- Spill cleanup material
- Emergency eyewash station
- Emergency care (911, PSS 574-3282)
- Emergency shower station
- Fire extinguisher
- Access to MSDS sheets for all chemicals used

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

- Abide by all guidance outlined in the Chemical Hygiene Plan (MCL-7702) and the Quality Assurance Plan (MCL-7701).
- Develop and encourage safe laboratory habits.
- Food will not be stored or consumed in lab areas.
- All work areas are to be kept clean and uncluttered.

- Safety glasses are required to be worn as posted.
- The appropriate personal protective equipment must be worn when required by the job.
- Report all accidents and near-miss accidents to your supervisor.
- All on-the-job injuries must be reported immediately to your supervisor.

4.2 Specific Hazards

These hazards have been identified by the MCLinc Hazard Matrix (see Attachment I). The following sections will list the hazard, its description, what the possible consequences are, and what controls are in place to mitigate the hazard. It is believed that the controls in place are adequate for all hazards identified in this section.

4.2.1 High Noise Level

This hazard is present when samples must be cut down to a smaller size. Cut-off saws used in this preparation can cause a high noise level. Potential consequences are hearing loss in personnel exposed to it. This hazard is controlled by personnel protective equipment, in the correct use of earplugs.

4.2.2 High Temperature

This hazard is associated with ovens and furnaces that may be used in sample preparation. The potential consequences are injury (burns) to personnel from contact with thermal components. The controls to mitigate this hazard include the guidance provided in the vendor manuals, the design of the equipment, and the method (experimental and instrumental design) by which the oven is used. The vendor manuals provide guidance for the proper usage and identification of hazards associated with this equipment. Insulated exterior surfaces and adequate sample transfer areas ensure no contact with hot surfaces occurs. Administrative controls include the labeling of furnaces as HOT and guidance provided in the vendor manuals and in the appropriate references in this document. PPE of thermal gloves, tongs, forceps, and the use of transfer vessels assist in handling hot materials.

4.2.3 Low Temperature

Exposure to low temperatures can occur during the transfer of liquid nitrogen from the transfer dewar to the cold stage or sample preparation. Potential consequences are frostbite or frostburn to personnel from direct contact liquid. This hazard is controlled by both administrative controls and personnel protective equipment (PPE). The administrative control is a guideline that provides the safety guidance and PPE requirements for the transfer of the liquid nitrogen. The PPE required for the transfer (face shield and cryo apron) provides adequate protection from the liquid nitrogen splash that may occur.

4.2.4 High Voltage

This hazard is common for some of the sample preparation equipment (although the majority of it is 110 volt). High voltage is required for instrument operations. Exposure to a high voltage source could lead to electric shock of personnel, destruction of equipment, and possible fire. This hazard is controlled by engineering and administrative controls. Engineering controls exist since these instruments have been manufactured to meet all safety and electrical codes. These instruments provide various safety interlocks to ensure that all sources of high voltage are properly shielded and that unintentional contact with high voltage sources is not possible. Administrative controls exist since maintenance of the equipment is covered by the vendor maintenance contracts. This provides specialized personnel to perform necessary maintenance. It is recognized that the highest risk from the high voltage can occur during non-routine operational conditions. These conditions are when a water leak is present at or near the instrument. Sources of water leaks can be water cooling lines for instrument or from drainage from the piping located above the ceiling panel in the room. Whenever uncontained water is detected, in conjunction with instrument operations, all operations should cease. This off-normal incident should be reported to the MCLinc Operations Manager.

4.2.5 High Pressure

High-pressure gas cylinders are used to provide valve control and support instrumental operations. Although the pressure used by the instrument is not high pressure (<150 psi) the gas cylinder itself represents a high pressure source. Failure of the gas cylinder could lead to asphyxiation or physical injury. This hazard is mitigated by the use of engineering, personnel protective equipment, and administrative controls. The engineering control in place includes a gas transfer buggy that has been designed to transport cylinders and a secure strapping device to keep the cylinder firmly in place after it has been unloaded from the dolly. The PPE includes the use of safety glasses in the case of a high-pressure release or component failure. The administrative control is the MCLinc policy for handling compressed gases. Reference Chemical Hygiene Plan for MCLinc (MCL-7702)

4.2.6 Off-shift Operations

Laboratory work being performed outside of normal shift may create a situation where backup support or help is not immediately available. This may lead to a lack or delay of emergency notification in case of an accident. This hazard is controlled by engineering controls and administrative controls. Engineering controls such as emergency pull boxes, telephones, fire sprinkler system, fire extinguisher and building public address system can be used to notify others of an off-normal situation. Administrative controls exist in that personnel are required to notify the PSS office (574-3282) if they will be occupying laboratory or office facilities during off-shift hours. This notification will help support the emergency response personnel in the event of an off-normal event. The performance of new (i.e. first time) activities are not permitted during off-shift hours. These controls and the use of training and on-the-job experience mitigate the hazards associated with this scenario.

4.2.7 Radioactive Materials

This hazard is common for sample preparation of radiological samples. Handling radioactive materials could lead to personnel exposure and/or contamination of equipment or property. This hazard is controlled by engineering, administrative and PPE controls. The engineering controls are the methods by which the samples are prepared. (All radioactive samples are prepared in a radiological area, in a hood, and are surveyed prior to removal from the area.) For proper analysis the sample has to be stable and firmly in place. The administrative controls are enacted by the HP organization.

4.2.8 Carcinogens

This hazard is common in the preparation of metallography samples. Handling of epoxy/hardeners in metallography samples could expose personnel to carcinogen vapors. This hazard is controlled by both engineering controls and administrative controls. The engineering controls are lab hoods for preparation. The administrative controls are small quantities.

This hazard could expose personnel to microscopic dust in the preparation of samples. High levels of microscopic dust could cause respiratory tract problems in personnel. This hazard is controlled by both engineering controls and personnel protective equipment (PPE). The engineering controls are lab hoods and the PPE controls are respirators.

4.2.9 Flammability

This hazard is associated with several of the chemicals (e.g. acetone, methanol, ethanol) that may be used during sample preparation. This hazard may cause a flash or burn to personnel and/or cause facility/equipment to ignite. The hazards associated with this are mitigated through the combination of engineering, administrative, and PPE controls. The engineering controls include lab hoods, secondary containment, small volume plastic squeeze bottles, fire extinguisher, and fire emergency pull boxes. All of these controls help to reduce the initial occurrence of a flammable incident as well as the spread of fire should a small laboratory fire occur. The administrative guidelines include MCLinc procedures/guidelines/and the use of small quantities of materials. Reference Chemical Hygiene Plan for MCLinc (MCL-7702). All of these controls help to reduce the possibility of an initial occurrence and severity of any occurrence that might happen. PPE controls to mitigate the hazard include the use of gloves and safety glasses.

4.2.10 Combustibility

This hazard is associated with several of the chemicals: (e.g. acetone, methanol, ethanol etc.) that may be used during sample preparation. These chemicals/vapors can combust when used around open flames or sparks. The hazards associated with these are mitigated through the combination of engineering and administrative controls. The engineering controls include lab hoods and strong ventilation of labs. The administrative control is the use of small volume of these chemicals.

4.2.11 Oxidizing or Reducing Ability

Description: corrosivity and adverse reactions. Oxidants may release oxygen and reductants may release hydrogen gas. Strong oxidizers (e.g., nitrate or perchlorate salts) can produce high gas pressure when heated, and may promote or support exothermic reactions or fires. Some compounds are shock-sensitive or explosive, and chemical incompatibilities may result in severe adverse reactions (e.g., mixing concentrated nitric acid with many organic compounds may cause a delayed explosion). Engineering controls are similar to those for flammability hazards. Administrative controls include familiarity with MSDS precautions for storage or use, and material substitution (e.g., clean glassware with detergent solutions, not chromic acid, etc.)

4.2.12 Acidity or Causticity

This hazard is associated with the use of acids or bases in the preparation of samples. This hazard could expose personnel to extreme pH materials that can cause burns and/or irritation. To mitigate this hazard engineering, administrative, and PPE controls are used. The engineering controls used include laboratory hoods, secondary containment, and laboratory clamps to hold the receiving vessel. These controls will help to minimize the chance of a spill and to contain any spill that may occur without exposing personnel. The administrative guidelines include the

use of MCLinc guidance and technical literature on the safe handling of chemicals. The Chemical Hygiene Plan for MCLinc (MCL-7702) helps to educate personnel as to the dangers associated with these materials. PPE controls include the use of safety glasses, tongs, face shields, rubber gloves, lab coats, and rubber aprons. These PPE controls help to prevent the exposure of personnel in the case of an accidental spill or splash.

4.2.13 Toxicity

This hazard is associated with the use of toxic materials in the preparation of samples. This hazard could expose personnel to toxic materials above accepted threshold values. To mitigate this hazard engineering, administrative, and PPE controls are used. The engineering controls used include laboratory hoods, secondary containment, and laboratory clamps to hold the receiving vessel. These controls will help to minimize the chance of a spill and to contain any spill that may occur without exposing personnel. The administrative guidelines include the use of MCLinc guidance and technical literature on the safe handling of chemicals. The Chemical Hygiene Plan for MCLinc (MCL-7702) helps to educate personnel as to the dangers associated with these materials. PPE controls include the use of safety glasses, face shields, rubber gloves, lab coats, and rubber aprons. These PPE controls help to prevent the exposure of personnel in the case of an accidental spill or splash.

4.3 Classified Work

Follow all guidance provided in the MCLinc Facility Security Plan (MCL-7706) for performing classified work.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

- Proper design.
- Start small--scale up if needed.
- Sample preparation - use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated shall be disposed of in accordance with MCLinc policies.

5.3 Environmental Risks

All chemicals are used in either small volumes, secondary containment, or closed systems for any open processes. No significant risks exist for the sample preparation activities performed by the MCLinc.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The method used to prepare a sample should be documented in the technical staff member's laboratory notebook. Documentation of the sample preparation methods will meet MCLinc and/or customer quality

7.0 REFERENCES

MCLinc *Industrial Hygiene Laboratory Quality Assurance Manual* (MCL-7719)

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Facility Security Plan* (MCL-7706)

Appendix I - Hazard Evaluation

MCLinc Hazard Evaluation		
ACTIVITY: SAMPLE PREPARATION (Non-asbestos) **LOCATION:** Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108 **EVALUATOR:** D.P. Hoffmann **DATE OF EVAL.:** April 23, 1998		
Potential Hazard	**Exist?**	**Description/Mitigation**
High Noise Level	YES	**DESCRIPTION:** Cut-off saws used in sample preparation can cause a high noise level. **POTENTIAL CONSEQUENCES:** Personnel exposed to high noise levels can cause hearing loss. **CONTROLS TO MITIGATE HAZARD:** Personnel protective equipment (e.g. ear plugs). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Temp. (≥250°C)	YES	**DESCRIPTION:** Ovens and/or muffle furnaces used to prepare samples may produce high temperature material handling hazards. **POTENTIAL CONSEQUENCES:** Burn to personnel from handling hot materials. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. oven design, temperature read-out), administrative controls (e.g. signs, guidelines, vendor manuals), and personnel protective equipment (e.g. gloves, tongs, hot pads).

MCLinc Hazard Evaluation

ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
LOCATION: Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108
EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		ADEQUACY OF CONTROLS: 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Low Temp.	YES	**DESCRIPTION:** Liquid Nitrogen (LN) is a cryogenic material required for operation of instruments. **POTENTIAL CONSEQUENCES:** Exposure of bare skin to (LN) can cause serious frostbite and freeze burns. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. Transfer dewar), personnel protective equipment (e.g. full-face shield, insulated gloves, cryo-apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Voltage (≥220V)	YES	**DESCRIPTION:** Exposure to high voltage source required for operation of instrument. **POTENTIAL CONSEQUENCES:** Electrical shock, burn or electrocution of personnel. Damage to equipment or facilities due to electrical discharge.

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ACTIVITY: SAMPLE PREPARATION (Non-asbestos)
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EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		CONTROLS TO MITIGATE HAZARD: Engineering controls (e.g. instrument design, safety interlocks), administrative controls (e.g. signs, guidelines, vendor manuals, lock out/tag out). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
High Pressure (≥150 psi)	YES	**DESCRIPTION:** Catastrophic rupture (most likely to valve stem) of compressed gas cylinders. **POTENTIAL CONSEQUENCES:** Rapid release of compressed gas could cause an asphyxiating environment or projectiles/fragments from rupture could injure personnel. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. gas moving cart, tie down straps), administrative controls (guidelines, MCL/MRI procedures). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Electromagnetic	NO	N/A
Moving Heavy Items	NO	N/A

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EVALUATOR:	D.P. Hoffmann	
DATE OF EVAL.:	April 23, 1998	
Potential Hazard	**Exist?**	**Description/Mitigation**
Machine Guarding	NO	N/A
Off-shift Operations	YES	**DESCRIPTION:** Lack of emergency notification support in the case of an accident. **POTENTIAL CONSEQUENCES:** Personnel or emergency situations may not be found/noticed for extended periods of times which may intensify the extent of damage. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. emergency pull boxes, telephone, fire sprinkler system), administrative controls (e.g. guidelines, MCLinc policy). **ADEQUACY OF CONTROLS:** 1 - controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Radioactive Materials	YES	**DESCRIPTION:** Handling of radioactive materials during sample preparation may expose personnel to radiation above ALARA levels. **POTENTIAL CONSEQUENCES:** Personnel may receive exposure to radiation greater than permissible levels. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. HEPA filtered hoods, frisking stations, radiological control areas), administrative controls (e.g. guidelines, MCLinc HP policies, Bioassay program, handle only low-level materials), personnel protective equipment

MCLinc Hazard Evaluation

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EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		(e.g. lab coats, shoe scuffs, gloves, safety glasses). **ADEQUACY OF CONTROLS:** 1- controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Confined Space	NO	N/A
Elevated Working Surface	NO	N/A
Hoisting and Rigging	NO	N/A
Carcinogens	YES	**DESCRIPTION:** Exposure to carcinogen vapors. **POTENTIAL CONSEQUENCES:** Handling of epoxy/hardeners during metallography sample preparation may expose personnel to vapors. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e g. lab hoods), administrative controls (small quantities). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Fibrous Materials	YES	**DESCRIPTION:** Handling of fibrous materials during

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EVALUATOR: D.P. Hoffmann
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Potential Hazard	Exist?	Description/Mitigation
		sample preparation could expose personnel to microscopic dust. **POTENTIAL CONSEQUENCES:** Personnel may receive high levels of dust. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hoods, respirators). **ADEQUACY OF CONTROLS:** 1 - controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Explosive Mixtures	NO	N/A
Glove box operations	NO	N/A
Ability to self-polymerize	NO	N/A
Shock sensitivity	NO	N/A
Thermal instability	NO	N/A
Rearranging Ability	NO	N/A
Pyrophoricity	NO	N/A
Flammability	YES	**DESCRIPTION:** Fire or flash from flammable materials. **POTENTIAL CONSEQUENCES:** Personnel burns or ignition of facility/equipment.

MCLinc Hazard Evaluation

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EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		CONTROLS TO MITIGATE HAZARD: Engineering controls (e.g. lab hoods, plastic squeeze bottles, fire extinguisher, fire protection systems, fire emergency pull boxes), administrative controls (e.g. guidelines, no open flames, small quantities), personnel protective equipment (gloves, safety glasses, lab coats). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Combustibility	YES	**DESCRIPTION:** Combustibility from flammable materials. **POTENTIAL CONSEQUENCES:** Personnel injury or destruction of facility/ equipment. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hoods), administrative controls (no open flames, small quantities, safety team), personnel protective equipment (shields, safety glasses, gloves). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.

MCLinc Hazard Evaluation

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EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
Peroxidizing Ability	NO	N/A
Water reactivity	NO	N/A
Oxidizing or reducing ability	YES	**DESCRIPTION:** Corrosivity and adverse reactions. Oxidants may release oxygen and reductants may release hydrogen gas. **POTENTIAL CONSEQUENCES:** Equipment corrosion or heat/pressure damage; personnel injury (burns or injuries from projected articles). **CONTROLS TO MITIGATE HAZARD:** Engineering controls are similar to those for flammability hazards. Administrative controls include familiarity with MSDS precautions for storage or use, and material substitution (e.g., clean glassware with detergent solutions, not chromic acid, etc.). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Acidity or causticity	YES	**DESCRIPTION:** Exposure to materials with high or low pH. **POTENTIAL CONSEQUENCES:** Chemical burn or skin irritation to personnel. **CONTROLS TO MITIGATE HAZARD:** Engineering

MCLinc Hazard Evaluation

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EVALUATOR: D.P. Hoffmann
DATE OF EVAL.: April 23, 1998

Potential Hazard	Exist?	Description/Mitigation
		controls (e.g. lab hood, vessel clamping/holding systems), administrative controls (e.g. guidelines, technical literature), personnel protective equipment (e.g. gloves, face shield, tongs, safety glasses, rubber apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate. **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Toxicity	YES	**DESCRIPTION:** Exposure to toxic materials. **POTENTIAL CONSEQUENCES:** Chemical exposure of personnel to toxic materials. **CONTROLS TO MITIGATE HAZARD:** Engineering controls (e.g. lab hood, vessel clamping/holding systems), administrative controls (e.g. guidelines, technical literature), personnel protective equipment (e.g. gloves, face shield, safety glasses, rubber apron). **ADEQUACY OF CONTROLS:** 1 - the controls are completely adequate. **SAFE TO OPERATE WITH CONTROLS:** YES **CORRECTIVE ACTION NEEDED:** None.
Increased reactivity	NO	N/A

MCLinc Hazard Evaluation		
ACTIVITY:	SAMPLE PREPARATION (Non-asbestos)	
LOCATION:	Rms. A102,A104,A106,B104,D101,D103,D105,D109,E100,E101,E102, E103,E104,E105,E108	
EVALUATOR:	D.P. Hoffmann	
DATE OF EVAL.:	April 23, 1998	
Potential Hazard	**Exist?**	**Description/Mitigation**
Ionizing Radiation	NO	N/A

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Materials and Chemistry Laboratory, Inc. Standard Operating Procedure		
Operation Guide X-Ray Diffraction: Materials and Chemistry Laboratory, Inc.:	Approved: MCLinc President	2/24/09 Date
	Quality Assurance Officer	2/24/09 Date

1.0 PURPOSE

This document and the documents referenced herein provide a framework for the safe and consistent operation of the x-ray diffractometer (XRD). It is accepted that operating personnel have an understanding of the instrumentation and theory of operation. This guideline will identify the hazards associated with the operation and ensure the safe usage of the instrumentation. This guideline will provide a high level of confidence in the results obtained and provide the foundation for quality control and quality assurance.

2.0 ROLES AND REFERENCES

2.1 Responsibilities

2.1.1 Principle Operator

The principle operator is responsible for the routine operation, upkeep of the instrumentation, and work area associated with the instrumentation. The appointment of the principle operator for each instrument is made by the *Chief Operating Officer*.

2.1.2 Secondary Operator

The secondary operator should be able to assist the primary operator in routine operation and maintenance. The secondary operator may be able to perform all operations at the same level of expertise as the primary operator, but this is not a requirement. Secondary operators may be certified by either the *Chief Operating Officer* or the principle operator.

2.1.3 *Chief Operating Officer*

The *Chief Operating Officer* represents the first level of line management which is responsible for supplying the resources for proper upkeep of the required instrumentation.

3.0 EQUIPMENT AND MATERIALS

3.1. Major Components

This table lists the major equipment covered by this guideline. The property number is the property number associated with the main instrument component. It is recognized that additional property numbers may exist for accessories and other secondary components.

Manufacture	Model #	Property #	Room #	Principle Operator	Secondary Operator
Philips Electronics	XRG-3100	K308903	D103	*M.R. Colberg*	*K.R. Tate*

3.2. Basic Process Description

X-ray diffraction measures the intensity of x-rays (i.e. Cu Kα) that diffract off a powder sample at discrete angles. The relative angle-intensity relationship provides crystallographic information about the sample. The diffraction pattern serves as a "fingerprint" of the phases of crystalline species present. The following is a brief overview of typical operational aspects of the instrumentation:

- The water cooled x-ray tube operates at high power (2200 watts maximum). Typical operating conditions are 40 kV and 35 mA (e.g. 1400 watts).
- XRD operates at atmospheric conditions.
- X-ray yield is contained/shielded by the instrument.

3.3. Laboratory Supplies

This non-inclusive listing provides a baseline for the types of supplies as well as engineering and administrative controls that should be available, as needed, to ensure a safe (personnel and environmental) work place.

- Disposable gloves
- Disposable laboratory waste bags
- Protective eye wear (during maintenance)
- Spill cleanup material
- Emergency eyewash station
- Emergency shower station

- Fire extinguisher
- Access to MSDS sheets for all chemicals used

3.4 Standards

The following component should be available for quality control and performance evaluation of XRD. The selection and use of this particular standard is based upon operator preference. The standard used should be documented in the appropriate logbook.

- Position/Intensity/Resolution Standard:
- Quartz (Supplied by Philips Electronic Instruments)

4.0 SAFETY PRECAUTIONS

4.1 General Laboratory Safety

- Abide all guidance outlined in the Chemical Hygiene Plan (MCL-CHP-001) and the Quality Assurance Plan (MCL-QAP-001).
- Develop and encourage safe laboratory habits.
- Food will not be stored or consumed in lab areas.
- All work areas are to be kept clean and uncluttered.
- Safety glasses are required to be worn as posted.
- The appropriate personal protective equipment must be worn when required by the job.
- Report all accidents and near-miss accidents to your supervisor.
- All on-the-job injuries must be reported immediately.

4.2 Specific Hazards

These hazards have been identified by the MCLinc. The following sections will list the hazard, its description, what the possible consequences are, and what controls are in place to mitigate the hazard. It is believed that the controls in place are adequate for all hazards identified in this section.

4.2.1 High Voltage

High voltage is required for instrument operations. Exposure to a high voltage source could lead to electric shock of personnel, destruction of equipment, and possible fire. This hazard is controlled by engineering and administrative controls. Engineering controls exist since these instruments have been manufactured to meet all safety and electrical codes. These instruments provide various safety interlocks to ensure that all sources of high voltage are properly shielded and that unintentional contact with high voltage sources is not possible. Administrative control exists since maintenance of the equipment is covered by maintenance agreements with the vendor. This provides highly specialized and skilled personnel to perform all necessary maintenance. These maintenance sub-contractors are also monitored and made to comply with all MCLinc safety rules and regulations. It is recognized that the highest risk from the high voltage can occur during non-routine operational conditions. These conditions are when a water leak is present at or near the instrument. Sources of water leaks can be water-cooling lines for the x-ray source or from drainage from the piping located above the ceiling panel in the room. Whenever uncontained water is detected all operations should cease. This off-normal incident should be reported to the MCLinc Manager.

4.2.2 Off-shift Operations

Laboratory work being performed outside of normal shift may create a situation where backup support or help is not immediately available. This may lead to a lack or delay of emergency notification in case of an accident. This hazard is controlled by engineering controls and administrative controls. Engineering controls such as emergency pull boxes, telephones, fire sprinkler system, fire extinguisher, building public address system can be used to notify others that of an off-normal situation. Administrative controls exist in that personnel are required to notify the PSS office (574-3282) if they will be occupying laboratory or office facilities during off-shift hours. This notification will help support the emergency response personnel in the event of an off-normal event. The performance of new (i.e. first time) activities is not permitted during off-shift hours. These controls and the use of training and on-the-job experience mitigate the hazards associated with this scenario.

4.2.3 Radioactive Materials

The possibility exists that the samples that are being analyzed will be radioactive. (See MCL-7710 for guidance on sample preparation.) Handling radioactive materials could lead to personnel exposure and/or contamination of equipment/property. This hazard is controlled by engineering, administrative and PPE controls. The engineering controls are the methods by which the samples are prepared. (All RAD samples are prepared in a radiological area and are surveyed prior to removal from the area.) The sample is firmly secured onto the sample platform. For proper analysis the sample has to be stable and firmly in place. The administrative controls are enacted by the HP organization. The HP technician must first establish a radioactive materials storage area (RMSA). This involves the survey of the instrument before and after the sample has been analyzed and the surrounding area. This HP support ensures that radiological material has not come loose during the analysis. The PPE containment involves the use of a

sample transfer box from the radiological area to the temporary RMSA at the instrument, the use of gloves while handling the sample, the skirting of the instrument area with yellow (RAD) plastic or tyvek, and radiological disposal of all PPE and lab supplies used in the RMSA.

4.2.4 Ionizing Radiation

This hazard recognizes the fact that x-rays are produced by the x-ray tube. These x-rays could be a potential source for personnel exposure. This hazard is mitigated by engineering controls. The basic requirements for the production of x-rays require the safety interlock system of the instrument to be operational. This instrument is surveyed for x-ray leakage. This means that it is not possible for a person to place their hand in, at, or near the source of x-ray production. It is also recognized that the source of the ionizing radiation can be totally removed by shutting off the x-ray gun.

4.3 Classified Work

Follow all guidance provided in the MCLinc Facility Security Plan (MCL-7706) for performing classified work.

4.4 Emergency Shutdown

The safest, most direct method of shutting the instrument off should be posted in clear plain sight on the front of the instrument. The instructions should be in large print, signed, dated, and laminated.

5.0 ENVIRONMENTAL AND WASTE MANAGEMENT CONCERNS

5.1 Waste Minimization Methods

Sample preparation — use of smallest possible beaker or test tube for cleaning samples or equipment (e.g. tweezers, spatula). Use only a portion of a paper towel or wipe as needed.

5.2 Waste Disposal Methods

All RCRA/TSCA/RAD waste generated by this process shall be disposed of in accordance with MCLinc policies (MCLinc Chemical Hygiene Plan, MCL-7702).

5.3 Environmental Risks

No appreciable environmental risks are noted at this time for XRD operation.

6.0 QUALITY AND PERFORMANCE DOCUMENTATION

6.1 Quality Assurance Documentation

The following information shall be documented in the time period stated. This information will provide direct documentation of the performance (calibration) parameters affecting the quality of the output (results) of the instrumentation. The documents resulting from these QA procedures will be kept in room *A108*.

Diffraction Calibration (monthly): A series of peaks from 20 to 90 degrees two theta will be used to measure position and linearity of the goniometer. A regression analysis will be performed on the resulting diffractogram. A more detailed discussion can be found in Appendix A.

Diffraction Resolution (monthly): A plot of the degrees two theta range from 65 to 70 will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is in Appendix A.

Detector Performance - (monthly): A series of low and high angle diffraction peaks will be used to track any variation in peak intensity with time. An example of this tracking is in Appendix A.

6.2 Performance Documentation

The following information shall be documented on the time period stated and after shutdown periods and maintenance. This information will document the scheduled maintenance, non-scheduled maintenance, and root cause for the instrumental non-conformance. The documents resulting from these performance procedures will be kept in room *A108*.

Scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or work which has been provided by the vendor in response to questions or operational abnormalities that is not clearly documented in the vendor's paperwork should be documented and attached to the vendor's paperwork.

Non-scheduled instrument maintenance (per event): A copy of the paper work provided by the vendor should be kept in chronological order. Any information or system work which is not clearly documented on the vendor's paperwork or work instructions provided over the telephone should be documented.

Instrument calibration non-conformance (per event): The actions required to bring the instrument back into compliance with operating specifications as noted in section 6.1 should be documented.

6.3 Vendor Manuals

Vendor manuals form the basis of the documentation for operating information. These manuals in combination with vendor/professional training and on-the-job training should allow the principle operator to safely, properly, and fully operate the instrumentation.

Vendor manuals shall be kept in good condition and be readily available during instrument operation.

6.4 Data Tracking

Diffraction patterns collected should be stored on disk in the raw data format.
All diffraction patterns should be given a unique filename. The file name should be logged with information concerning the sample ID number, the operating conditions, the disk storage ID number, and the date.

7.0 REFERENCES

MCLinc *Chemical Hygiene Plan* (MCL-7702)

MCLinc *Quality Assurance* (MCL-7701)

MCLinc *Sample Preparation Guide* (MCL-7710)

Appendix A
Quality Assurance Documentation

Quality assurance documentation for the XRD is obtained on a monthly basis. The quartz standard from Philips Electronic Instruments should be run at 40 k*V* and *35 mA*. The program used to do the standard run covers the range 20 to 90 degrees two theta in 0.01 degree steps. Each step is counted for 2.5 seconds and the peak location is calculated by the XRD computer using the centroid second derivative technique. The plots of the diffractogram and the computer printout are archived in Room *A108*. Subsequent computer analysis of the data is done using Microsoft Excel.

There are three areas of calibration interest. The first of these is the two theta position of the diffractometer. To evaluate this parameter of XRD operation a series of peaks encompassing the general range from 20 to 90 degrees two theta are compared to the standard quartz profile. The differences between the monthly run and the standard peak locations are tabulated and and a linear regression analysis is performed on the diffractogram results (Figure 1). The consistency of the slope and intercept from the linear regression is tracked. Should the regression analysis show inconsistency, the root cause will be determined and actions will be taken to correct the inconsistency.

The second area of interest is the check of x-ray tube and detector performance. A series of low and high angle diffraction peaks is used to tract any variation in peak intensity. An example of this tracking is shown in Figure 3. If the intensity falls below an acceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

The third area of interest is the check of detector performance. A plot of the 65 to 70 degrees two theta region versus intensity will be observed for the split of the five peaks that make up this region of the quartz standard diffractogram. An example of this region is shown in Figure 2. In addition, the Ka1 peak FWHM at 59.2 degrees two theta will be determined and plotted for variation. If the resolution increases to an unacceptable level, the root cause will be determined and actions will be taken to correct the variance from acceptable operating conditions.

Figure 1
Linear Regression Analysis Plot

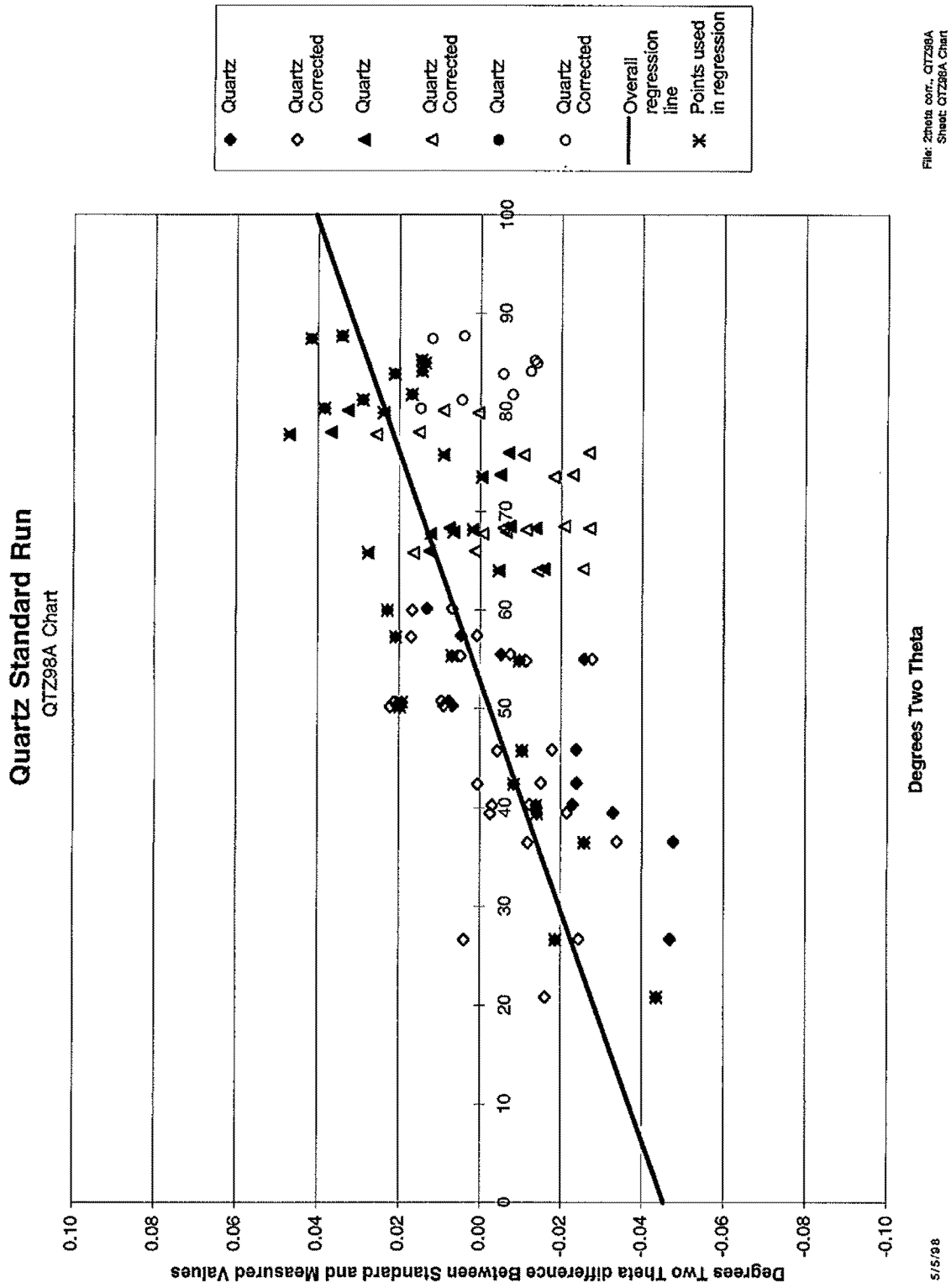


Figure 2
Example of Resolution Check

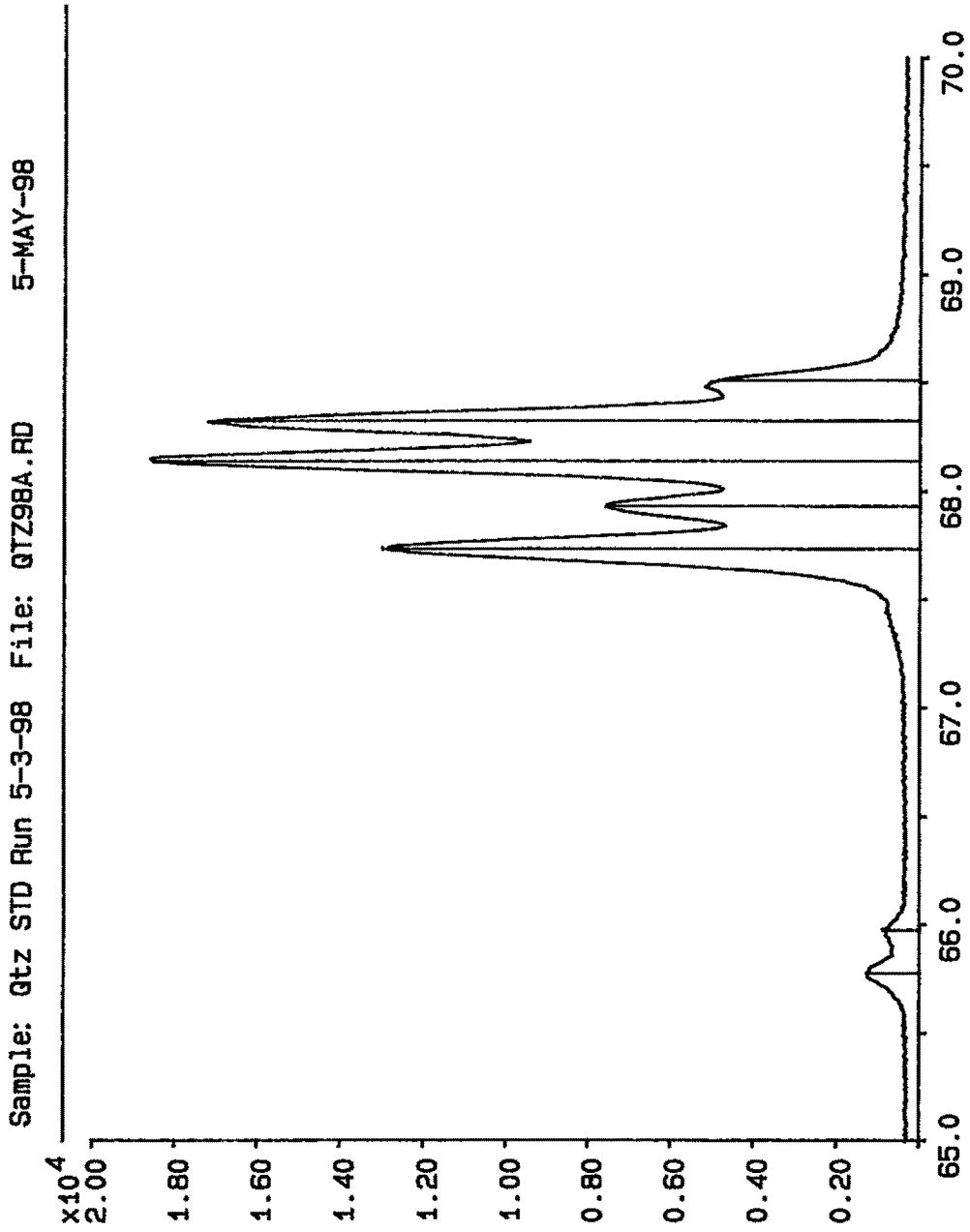
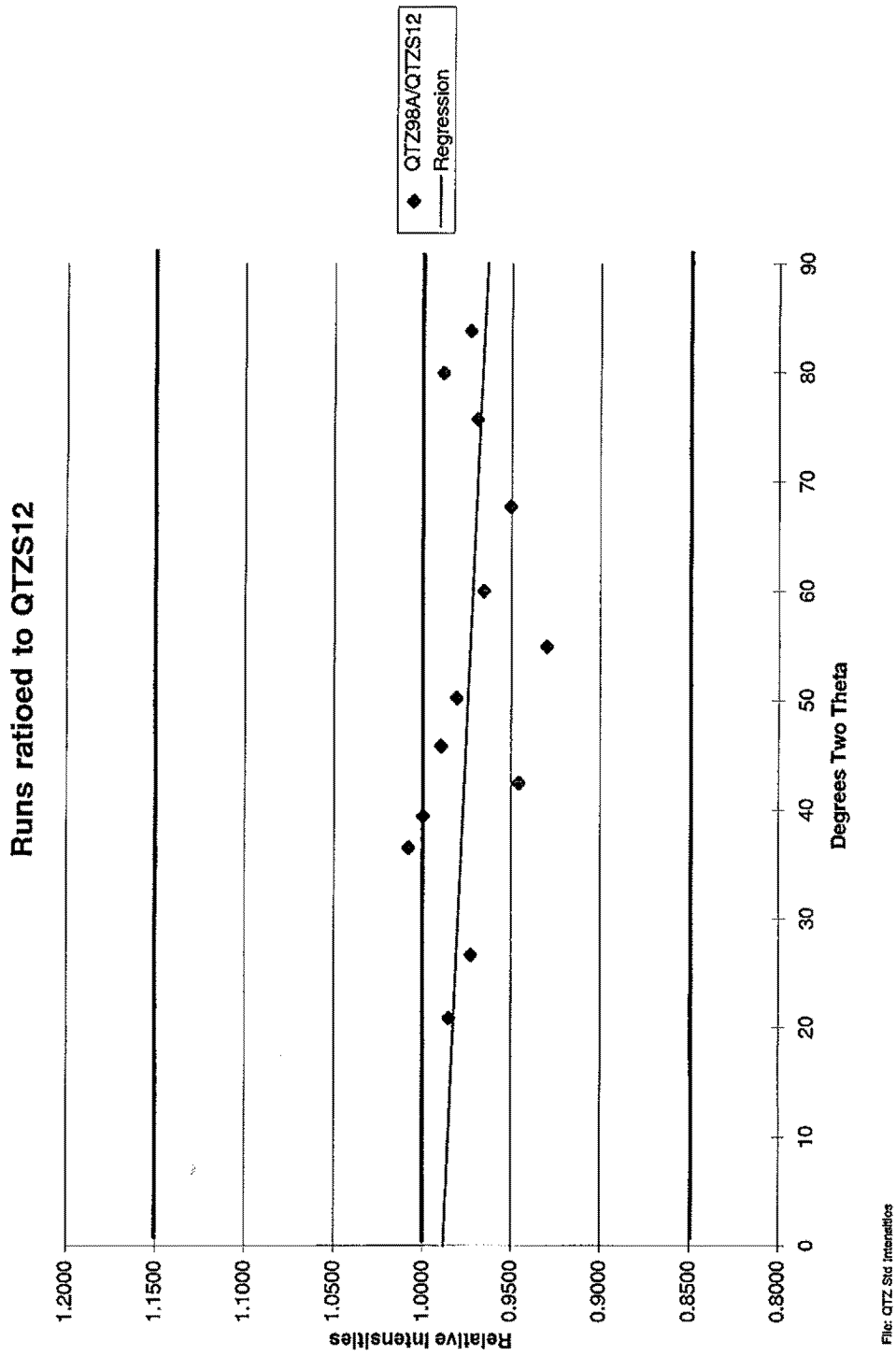


Figure 3
Example of Quality Assurance Intensity Check



UNCONTROLLED
INFORMATIONAL USE ONLY

MATERIALS AND CHEMISTRY LABORATORY, INC.
STANDARD OPERATING PROCEDURE

Determination of Uranium by a Modified Davies-Gray Titration: Materials and Chemistry Laboratory, Inc.	Approved: MCLinc President	9/15/11 Date
	Quality Assurance Officer	9/12/11 Date

1.0 PURPOSE

This procedure applies to samples of uranium compounds of the nature UxFz,, UxOyFz, UxOy and others relating to uranium contaminated scrap materials where interfering elements are kept to a minimum.

2.0 SCOPE

This procedure may also be applied to determine levels of uranium in aqueous and solid samples.

3.0 ROLES AND RESPONSIBILITIES

MCLinc analyst is responsible for performing the analysis on the samples per this procedure, reviewing the results, and reporting any problems.

The Operations Manager or Project Manager represents the first level of management and provides project oversight.

4.0 MATERIALS AND APPARATUS

- Platinum wire: 12"
- Orion Ag-AgCl Half-Cell Single Junction Reference Electrode
- *Thermo Scientific Orion Star pH/ISE Benchtop Meter* or equivalent
- Micro buret-2 ml, 0.002 ml graduations, Gilmont GS-1200A
- Magnetic stirrer
- Teflon coated stir bars
- Hot plate
- Fume hood
- Muffle or tube furnace

- Assorted laboratory glassware – cleaned in laboratory detergent solution and rinsed well in DI H_20
- Platinum or quartz boats
- Thermometer

5.0 REAGENTS

- Sulfuric acid (H_2SO_4): 96%, concentrated.
- Sulfamic acid (H_2NSO_3H): reagent grade.
- Phosphoric acid (H_3PO_4): 85%, concentrated.
- Ferrous sulfate ($FeSO_4.7H_2O$)), granular or crystal.
- Nitric acid (HNO_3): 70%, concentrated.
- Ammonium molybdate [$(NH_4)_6Mo_7O_{24}.4H_2O$], crystals.
- Vanadyl sulfate ($VOSO_4.nH_2O$), 99% pure.
- Potassium dichromate ($K_2Cr_2O_7$), Primary Standard Grade.
- Triuranium octaoxide, (U_3O_8), highly pure.
- Distilled or deionized water.
- Chromic acid for glassware cleaning.
- Sodium hydroxide, (NaOH), pellets or other caustic chemical for acid neutralization
- Laboratory detergent.

6.0 REAGENT PREPARATION

A. <u>1 M Sulfuric acid solution</u>:

1. To a 2-liter volumetric flask, add ~ 1000 ml DI H_2O.
2. Carefully, while holding flask under the cold water faucet, add 110 ml of concentrated H_2SO_4 while swirling.
3. Allow to cool and then dilute to volume with DI H_2O.

Shelf life: ~ 6 months.

B. <u>1.5 M Sulfamic acid</u>:

1. To a 1-liter volumetric flask, add 145.5 g of sulfamic acid and ~ 800 ml of DI H_2O.
2. Stir on a magnetic stirrer with gentle heat sufficient to dissolve the solids.
3. Cool and dilute to volume with DI H_2O.

Note: 100 ml of this solution is used in the preparation of the reagent in 6.0 D.

Shelf life: ~ 6 months.

C. 1 M Ferrous sulfate solution:

1. To a 100 ml volumetric flask, add ~65 ml DI H_2O.
2. Carefully add 10 ml of concentrated H_2SO_4.
3. Add 28 g of $FeSO_4.7H_2O$.
4. Carefully shake to dissolve, cool, and dilute to volume with DI H_2O

Shelf life: 2 days

D. Nitric-sulfamic acid solution with ammonium molybdate:

1. To a 1 liter storage bottle, add 400 ml of DI H_2O.
2. Add 4.0 g of $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ and dissolve.
3. Add 500 ml of concentrated HNO_3 and mix.
4. Add 100 ml of the sulfamic acid solution previously prepared in 6.0 B and mix well.

Shelf life: ~ 6 months

E. 0.027 N Potassium dichromate standard solution:

1. Dry *NIST* SRM 136*F* $K_2Cr_2O_7$ or equivalent primary standard grade for *2* hrs at 110 °C.
2. Cool in dessicator.
3. Weigh out about 1.325 g (accurately to 4 decimal places) of the dichromate for 1 liter of solution corrected for the assay.
4. Add the weighed dichromate to a 1-liter volumetric flask (calibrated), dissolve and dilute to volume with DI H_2O.
5. Transfer the solution to a 1 liter glass bottle.
6. Calculate the normality of the solution.

Normality ($K_2Cr_2O_7$) = mass(g) x assay x 1 mol/294.1844 g x 6 eq/mol x 1/vol. flask (L)

Shelf life: indefinite.

F. 0.008 N Potassium dichromate solution:

1. Dissolve 0.39 g $K_2Cr_2O_7$ in a 1-liter volumetric flask with DI H_20.
2. Bring to volume with DI H_2O.
3. This solution is used to oxidize impurities in the phosphoric acid, its normality does not have to be precise.

Shelf life: indefinite

G. Uranium standard solution, ~ 340 ppm U in 10% HNO_3.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.4 g of U_3O_8 in a 200 ml tall form beaker.
5. Add 50 ml 10% HNO_3 and 50 ml conc. HNO_3.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 1 liter calibrated volumetric flask using DI H_2O.
8. Add 45 ml of conc. HNO_3.
9. Dilute to volume with DI H_2O.
10. Transfer the solution to a 1 liter glass bottle.

Shelf life: indefinite

Calculation of uranium concentration in the standard solution.:

0.4000 g U_3O_8/1 L x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 339.09 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

H. Uranium standard solution, ~ 42 ppm U in concentrated H_3PO_4.

1. Place a small quantity of NBS 950b U_3O_8 in a quartz or platinum boat.
2. Insert the boat into the tube furnace at 800 deg C for 1 h.
3. Cool in a dessicator to room temperature.
4. Weigh out ~0.05 g of U_3O_8 in a 200 ml tall form beaker.
5. Add ~25 ml concentrated H_3PO_4.
6. Heat gently on a hot plate to dissolve.
7. Cool and transfer to a 100 ml volumetric flask using concentrated H_3PO_4.
8. Dilute to volume with concentrated H_3PO_4.

Shelf life: U+4: Analyze within a week, total U indefinite

Calculation of uranium concentration in the standard solution.:

0.0500 g U_3O_8 x 0.99968 g U_3O_8/1.00000 g U_3O_8 x 0.848001 g U/ 1.000000 g U_3O_8 x 1000 mg U/1 g U = 42.386 mg U/L

Standards are reanalyzed when the deviation from the accepted value exceeds 0.1 mg.

7.0 PROCEDURE

A. Total U for samples dissolved in dilute HNO_3

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 40 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 3 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

B. Total U for samples dissolved in concentrated H_3PO_4.

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).
   c) 3 ml conc. H_2SO_4 and swirl.
   d) 5 ml 1.5 M sulfamic acid and swirl.
   e) 28 ml conc. H_3PO_4 down the beaker walls and swirl.
   f) 11 ml DI H_2O and swirl.
   g) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   h) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   i) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   j) 100 ml 1 M H_2SO_4 (wash down thermometer).
   k) 100 mg – 120 mg vanadyl sulfate.

2. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on uranium concentration, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

3. Place the remaining solution in the appropriate waste container.

C. Procedure for U^{+4} (sample must be dissolved in concentrated H_3PO_4)

1. To 300 ml tall form beaker add the following in order:
   a) Magnetic stir bar.
   b) 15 ml sample (pipetted).

Allow the beaker to stand while the rest of the reagents are added to a separate clean beaker.

2. To a separate clean 250 ml beaker, add:
   a) 15 ml conc. H_3PO_4 and swirl.
   b) 3 ml conc. H_2SO_4 and swirl.
   c) 5 ml 1.5 M sulfamic acid and swirl.
   d) 13 ml conc. H_3PO_4 down the beaker walls and swirl.
   e) 11 ml DI H_2O and swirl.
   f) 1 ml 0.008 N $K_2Cr_2O_7$ and swirl.
   g) 5 ml 1 M $FeSO_4$ and swirl. (Allow 30-60s reaction time, adjust temperature to 40-43 deg C during this time period).
   h) 10 ml nitric-sulfamic acid solution and swirl. (Allow 3 min reaction time, weigh out vanadyl sulfate and prepare electrodes during this time period).
   i) 100 ml 1 M H_2SO_4 (wash down thermometer)..
   j) Add this solution to the beaker containing the pipetted sample (steps 1a-1b).
   k) Add 100 mg – 120 mg vanadyl sulfate.

3. Insert the electrodes and immediately titrate with 0.027 N $K_2Cr_2O_7$. (The endpoint is between 590-620 mv.) Rapidly add titrant until ~520 mv is reached. Then add titrant in 0.01 ml or 0.002 ml increments depending on sample size, and record the potential at each addition of titrant. Use the second derivative method of calculating the endpoint.)

4. Place the remaining solution in the appropriate waste container.

8.0 CALCULATIONS

Sample Titration Data showing 2nd derivative method:

A Volume (ml)	B Potential (mV)	C d(B)/ d(A)	D $d^2(B)/$ $d^2(A)$
0.000	559		
0.008	576		
0.010	583		
		\	
		7,500	
		/ \	
0.012	598		+ 4,500
		\ /	
		12,000	
		/ \	
0.014	622		- 2,500
		\ /	
		9,500	
		/	
0.016	641		

Endpoint = 0.012 ml + 0.002 ml [4,500 / (4,500 + 2,500)] = 0.01329 ml

Sample Calculation for the Amount of Uranium:

[0.01329 ml - 0.0024 ml (blank)] x 0.027039 meq/ml x 1 mmol/ 2 meq x 238.03 mg U/ 1 mmol U = 0.035 mg U

Reporting limit – The reporting limit for this procedure is the amount of uranium corresponding to 0.005 ml of titrant after blank correction.

9.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Because all materials utilized in this procedure are potentially radioactive sources, all samples, waste, and standards will be appropriately labeled and handled according to MCL-7718 and MCL-7715.

The waste will be minimized by using small volumes and minimizing quantities utilized for sample preparation and standards preparation. Materials for disposal will be segregated and properly labeled. Where possible, the waste will be reduced by known treatment methodologies.

Rad waste will be measured and documented and where necessary turned over to an approved commercial handling and disposal service.

10.0 REFERENCES

1. W. Davies and W. Gray, "A Rapid and Specific Titrimetric Method for the Precise Determination of Uranium Using Iron (II) Sulfate as Reductant,"Talanta" 11, (1964), p. 1203.

2. Eberle et al., "Titrimetric Determination of Uranium in Product, Fuel, and Scrap Materials After Ferrous Ion Reduction in Phosphoric Acid," New Brunswick Laboratory Progress Report No. 252, July, 1970.

3. R.J. Jarabek, Transport Measurements of UF_5 Using a Precision Analysis for U^{+4}, K/PS-5017, Martin Marietta Energy Systems, Inc., Oak Ridge Gaseous Diffusion Plant, April 2, 1984.

4. D.A. Skoog and D.M. West, Fundamentals of Analytical Chemistry, Holt, Reinhart, and Winston, Inc., pp. 550-554, 2nd ed., 1969.