# ROY F. WESTON, INC.







# SAMPLING & ANALYSIS PLAN

# Non-Time Critical Removal Action Causeway Phase I

Stratford Army Engine Plant - Stratford, Connecticut

Contract No. DAAD05-97-D-7004 Delivery Order No. 0187

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Prepared by

ROY F. WESTON, INC. 148 Eastern Boulevard Glastonbury, Connecticut 06033

# SAMPLING AND ANALYSIS PLAN APPROVALS

Non-Time Critical Removal Action Phase I Causeway Stratford Army Engine Plant Stratford, Connecticut

Contract Number: DAAD05-97-D-7004

# SAMPLING AND ANALYSIS PLAN APPROVALS

By their specific signature, the undersigned certify that this Sampling and Analysis Plan is approved for utilization during the Non-Time Critical Removal Action (Phase I Causeway) at the Stratford Army Engine Plant in Stratford, Connecticut.

WESTON – Project Manager John-Eric Andersson	Date
WESTON – Construction QC Manager Joseph Wasiuk	Date
WESTON – QC Officer	Date

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# 1. PROJECT DESCRIPTION

This Sampling and Analysis Plan (SAP) defines the sampling techniques and analytical protocols that will be employed by Roy F. Weston, Inc. (WESTON) during completion of the Non-Time Critical Removal Action (NCRA) Causeway Project (Phase I) located at the Stratford Army Engineering Plant (SAEP) site in Stratford, Connecticut.

#### 1.1 SITE DESCRIPTION

SAEP consists of approximately 124 acres, of which an estimated 76 acres are improved land consisting of 49 buildings, paved roadways and grounds, and five paved parking lots. Included in the improved land are an estimated 10 acres along the Housatonic. River where fill was placed over tidal sediments during the development of SAEP facility, including the Causeway. Riparian rights are associated with the remainder of the SAEP facility. A riparian right is a right of access to, or use of, the shore, bed, or water of land on the bank of a natural watercourse. The riparian rights property consists of intertidal flats of the Housatonic River. An estimated two acres of property compose the Causeway, constructed to provide access to the river channel.

The Causeway is an approximately two-acre portion of this fill area and was originally constructed to provide access to the river channel. The Causeway was initially constructed and used as a means of launching seaplanes in the 1930s. Additional materials of unknown origin were deposited along the northern edge of the Causeway during the 1950s and 1960s. The source of the fill used to construct the Causeway is unknown, but it has been found to contain soil, cobbles, and construction debris (e.g., concrete, brick, and asphalt). Smaller amounts of other material (e.g., wood, glass, cinders, ash, and rebar) were also observed during field investigation activities. It was also reported that paint solvents and wastes were burned on the Causeway as part of fire-training operations.

Presently, the Causeway is overgrown with small trees, shrubs, and grasses. The surface of the Causeway, including the tidal flats, contained scattered pies and outcroppings of oversized debris, primarily concrete, boulders, and asphalt). Two dirt access roads extend along the entire length of the Causeway. The only structures located on the Causeway include Building 59 (near the entrance to the Causeway) and the former boat ramp and weather station (near the Housatonic River channel).

#### 1.2 PROJECT DESCRIPTION

Access to the Causeway is controlled by a fence and gate and is further restricted by the existence of several buildings in the area. Phase I activities include the demolition of Building 59, Building 5, and the containment area adjacent to Building 34, to improve equipment access to the Causeway for Phase II construction.

Building 59, a concrete building with concrete blast walls, located near the origin of the Causeway from the facility, was constructed to house the nose cones of missiles, including the explosive charges used to open the nose cones. There is currently no unexploded ordnance present at the SAEP facility. There are two additional structures on the Causeway – a weather station constructed on a concrete slab, and a concrete boat ramp. These structures will be removed to within 2 feet of existing grade.

On the site proper, Building 5 and a bermed area adjacent to Building 34 will be demolished. Building 5, constructed of masonry block, has been found to contain asbestos-containing materials (ACM). Phase I activities include abatement of the ACM and collection of loose paint chips within Building 5, followed by demolition of



the building. The containment berm adjacent to Building 34 formerly housed an above-ground storage tank (AST) farm. Historically, the ASTs were removed, clean fill was placed over the tank supports within the containment berm, and the area covered with asphalt. Phase I activities include removal of the Building 34 containment berm, protective posts and former tank supports to grade. All demolition debris will be sampled and analyzed in order to characterize the materials for proper off-site disposal.

Previous investigations on the Causeway have identified soil containing volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), vanadium and zinc in excess of the CTDEP Remediation Standard Regulations (RSRs) Pollutant Mobility Criteria, ten times the Groundwater Protection Criteria, or ten times the federal Ambient Water Quality Criteria (AWQC). This soil will be excavated, containerized and transported to on-site staging areas for disposal characterization. An estimated 35 cubic yards of soil are to be excavated from six distinct areas on the Causeway. Confirmation sampling to verify that all soil containing contamination above the RSR and AWQC action levels has been removed will be conducted by Harding ESE (the Engineer) and is not the responsibility of WESTON. The limits of excavation will be established by the Contracting Officer's Representative.

During Phase I activities, oversized debris will be removed from the surface of the Causeway, cleaned of excess soil using high-pressure cleaning equipment, and shipped off-site for disposal. Washing will be performed in close proximity to the removal area in a manner such that wash waters infiltrate the Causeway soils, thereby minimizing the potential for migration of sediments.

WESTON will be required to containerize any liquids collected from staging areas and from the decontamination of equipment and supplies intended for reuse. These liquids will be sampled and analyzed for total VOCs. A Chemical Waste Treatment Plant is located in Building 63 capable of treating the majority of constituents anticipated in waste waters generated during this project. However, VOC concentrations in the Treatment Plant influent are limited to a maximum of 100 parts per billion (ppb). Therefore, the objective of the water sampling is to confirm compliance with the 100 ppb VOC influent limit prior to discharge to the Treatment Plant. In the event that VOC concentrations exceed 100 ppb, the liquid will be treated on-site using activated carbon until subsequent sampling results are below the influent limit.

# 1.3 PROJECT ACTIVITIES

The scope of services for Phase I include the following:

- Abandonment of one monitoring well and preservation of the remaining wells on the Causeway.
- Installation and maintenance of erosion and sedimentation controls around the work area.
- Clearing and chipping of trees and brush from the Causeway area.
- Installation and monitoring of heave platforms at the tidal flat surrounding the Causeway and completion of a topographic survey of the Causeway.
- Removal of the containment curbing and berm to match surrounding grade along with protective bollards followed by paving.
- Characterization, demolition and off-site disposal of Building No. 5, including utility disconnections, and asbestos abatement. The slab and foundation will remain in place.



- Excavation, characterization and off-site disposal of contaminated soil present at six locations on the Causeway. Post-excavation samples and data analysis will be performed by Harding.
- Characterization, demolition and off-site disposal of Building 59, the weather station, and boat ramp.
- Characterization, removal and off-site disposal of oversized surface debris viable at the surface of the Causeway including rinsing of soil prior to removal.
- Decontamination of equipment and supplies intended for reuse. Decontamination liquids are to be containerized, characterized and disposed at the on-site Chemical Waste Treatment Plant.

WESTON will be responsible for collecting characterization samples of the following anticipated waste streams: demolition debris, Causeway debris, Causeway soil and decontamination liquids.



# 2. PROJECT ORGANIZATION AND RESPONSIBILITIES

The project organization and individuals responsible for implementing the Quality Assurance (QA) aspects of the Sampling and Analysis Plan (SAP) for the work at SEAP is presented in Figure 2-1. Their responsibilities are indicated in the subsections that follow.

#### 2.1 PROJECT PERSONNEL

WESTON's management team will be led by the Program Director (Mr. Tony Riccio), the Regional Operations Manager (Mr. Todd Walles), and Project Manager (Mr. John-Eric Andersson). They will be responsible for WESTON's overall performance on this project. The WESTON project field team will consist of a Construction Quality Control (QC) Manager (Mr. Joseph Wasiuk), a Construction Superintendent (Mr. Steven O'Brien), a Quality Control (QC) Officer (Mr. Andy Harris), and a Site Health & Safety Coordinator (SHSC – Mr. Tim Laquerre). The management team will closely monitor site activity, performance, costs, schedule, QC, and safety to ensure that the project objectives set forth in the Specifications and described in Section 1 of the Work Plan are achieved.

## 2.1.1 Project Manager

The Project Manager will be responsible for resource planning, schedule coordination, and overall project administration of task-specific activities in accordance with the specifications.

# 2.1.2 Construction QC Manager

The Construction QC Manager is responsible for ensuring that the program is in compliance with the work plan, and will perform periodic audits to verify adherence of activities to the provisions of the SAP.

#### 2.1.3 Quality Control (QC) Officer

The QC Officer is responsible for ensuring the implementation of the SAP as it applies to field sampling and analysis processes for the site. Specifically, the QC Officer is responsible for overseeing the following during sampling activities:

- Proper sample container preparation and labeling.
- Sample preservation and transportation.
- Sample chain-of-custody.
- Proper sampling procedure (i.e., equipment calibrations).
- Sample identification.
- Field documentation.

# 2.1.4 Construction Superintendent

The Construction Superintendent (CS) reports to the Project Manager and is responsible for supervising field implementation of the project. The (CS) provides direct supervision of field staff and together with the Site Health & Safety Coordinator is responsible for ensuring that all personnel adhere to the requirements of the Site-Specific Safety and Health Plan (SSHP).



# 2.1.5 Site Health & Safety Coordinator

The SHSC is responsible for on-site implementation and enforcement of the SSHP, air monitoring, accident reporting, and overall site safety. The SHSC has the authority to halt any project phase or operation deemed either inherently dangerous to life and health, or not in compliance with the SSHP. In addition, the SHSC can remove from the project any person who is deemed inherently unsafe or a threat to the safety of other individuals at or in the vicinity of the project. The SHSC reports directly to the PM but receives technical oversight from the Program Safety Manager (Mr. George Crawford, CIH). The SHSC will supervise all of the field construction activities as described in the SOW. The SHSC will ensure that the remedial actions conform to the requirements of the Work Plan and the SSHP. The SHSC will also assist with sample collection.

## 2.1.6 Project Scientist

The Project Scientist will serve as the Sampling Officer and will coordinate all project sampling activities. The Project Scientist will perform a dual role as QC Officer. The sampling responsibilities will include:

- Daily implementation of the SAP.
- Documentation of deviations from the SAP, with explanation.
- Provide sampling instructions and oversight.
- Coordinate sampling activities with the off-site laboratory.
- Preliminary review of laboratory data.
- Establish a data tracking and management system.
- Assign sample identification number.
- Assign and direct sampling tasks to WESTON technicians.

#### 2.1.7 Field Personnel

Field personnel are responsible for sample collection, initiation of the chain of custody, and the shipment of the samples to the laboratory. All field personnel will have documented experience with the collection of hazardous waste samples and meet all health and safety requirements for this project.

#### 2.2 LABORATORY RESPONSIBILITIES

The laboratory analyses will be conducted by Mitkem Corporation, a USACE validated analytical laboratory located in Warwick, Rhode Island. The point of contact for the laboratory is Mr. Paul Senecal, Vice President, at (401) 732-3400. The laboratory responsibilities and staffing are detailed in the following subsections.

Laboratory QC procedures and responsibilities will be in accordance with this Plan and the analytical laboratory's CENAE-approved Quality Assurance Project Plan (QAPP). A copy of the Mitkem QAPP is attached.

# 2.2.1 Analytical Laboratory Manager

The Analytical Laboratory Manager is responsible for ensuring that all analytical tasks for this project are conducted according to the requirements of the SAP.



# 2.2.2 Laboratory Project Manager

The Laboratory Project Manager is responsible for scheduling project analytical requirements, monitoring analytical status/deadlines, approving laboratory reports, and coordinating data revisions/corrections and resubmitting packages to project staff. The Laboratory Project Manager will prepare/review analytical work and ensure that laboratory personnel understand and conform to the elements of the SAP that are related to their activities.

## 2.2.3 Laboratory QA Manager

The Laboratory Quality Assurance (QA) Manager will ensure conformance with authorized policies, procedures, and sound practices, and will recommend improvements as necessary. The Laboratory QA Manager will inform the Laboratory Project Manager of nonconformance to the SAP. In addition, the Laboratory QA Manager may approve laboratory data before reporting or transferring data to permanent storage and be responsible for maintaining supporting information and other performance indicators to demonstrate that the systems that produced the data were in control. The Laboratory QA Manager will also review results of internal QA audits and recommend corrective actions and schedules for their implementation.

# 2.2.4 Laboratory Chemists/Technicians

An effective laboratory QA program depends on the performance of all laboratory staff performing analyses. The responsibilities of laboratory chemists and technicians include:

- Performing initial review of QC data for acceptability.
- Recording data in bound laboratory notebooks.
- Informing direct supervisors of any problems with instruments or methods to ensure that prompt and effective corrective action is taken.



# 3. FIELD ACTIVITIES

Field sampling activities for the project consist of characterization sampling of soil, demolition and oversized debris, and decontamination fluid generated during Phase I activities.

#### 3.1 CHARACTERIZATION OF EXCAVATED SOIL

#### 3.1.1 Rationale

Samples of the excavated soil generated during Phase I activities will be collected, analyzed for waste characterization, and transported off-site for disposal. Used personal protective equipment (PPE) generated during the excavation of the contaminated soil will be disposed of with the soil and demolition debris.

The collection of additional waste characterization samples will be required if work procedures alter the waste stream or different disposal options are considered. The frequency of the samples will be determined by the disposal facility.

# 3.1.1.1 Background, QA/QC, and Blank Samples and Frequency

Background samples will not be collected. If the Contracting Officer (CO) determines that background samples are required to document existing site conditions prior to remedial action, samples will be collected by the Engineer. Field duplicate samples will be submitted as QC duplicates. The QC duplicates will be collected for each analytical parameter and will represent approximately 10% of the field samples collected during each sampling event (1 per every 10 samples).

#### 3.1.2 Procedures

#### 3.1.2.1 Sampling Procedures for Chemical Analyses

Samples of the soil will be collected to determine the off-site disposition of the material. The test parameters shall be selected to comply with all applicable Federal, State and local laws and regulations, and shall be identified in accordance with the accepting disposal facilities requirements. Due to the small anticipated quantities of soil, WESTON will collect one representative sample from the containerized soils upon completion of excavation of the six areas. The sample shall be transported to the laboratory within two days of collection.

# 3.1.2.2 Field Quality Control Sampling Procedures

## **Equipment Blank/Rinsate Blanks**

Field equipment blank/rinsate blanks will not be collected as WESTON anticipates utilizing dedicated equipment for the collection of soil samples.

#### **Duplicates**

Due to the small anticipated quantities of soil, a duplicate sample will not be collected for analysis. Rather, the representative sample will be of sufficient quantity to allow the laboratory to repeat any or all of the selected analyses in the event of data validity concerns.



## Matrix Spikes/Matrix Spike Duplicates Samples

Matrix Spike/Matrix Spike Duplicate, trip blanks, temperature blanks, equipment blanks, and rinsate blanks will not be collected for soil waste characterization samples.

#### 3.1.2.3 Decontamination Procedures

It is anticipated that only dedicated sampler jars, and disposable scoops will be used for sample collection of characterization samples. Any non-dedicated equipment used for sampling purposes will be decontaminated after each sample is collected in order to eliminate the possibility of cross-contamination between sampling locations. Decontamination of reusable equipment will consist of a non-phosphate laboratory detergent rinse (liquinox/water wash), potable water rinse, a double deionized water rinse, and air-drying. Decontamination fluids will be containerized for testing prior to on-site treatment and disposal.

#### 3.2 CHARACTERIZATION OF DEMOLITION AND OVERSIZED DEBRIS

## 3.2.1 Rationale/Sampling Locations

It is anticipated that several different debris types will be generated from building demolition and oversized debris removal activities including: concrete, metal, plastics, asphalt, etc. Demolition and oversized debris samples will be collected to determine the off-site disposition of the material. The test parameters for each debris type shall be selected to comply with all applicable Federal, State and local laws and regulations, and shall be identified in accordance with the accepting disposal facilities requirements. One composite sample shall be collected from each debris type in accordance with the requirements of the accepting facility. The collection of additional waste characterization samples will be required if work procedures alter the waste stream or different disposal options are considered. The frequency of the samples will be determined by the disposal facility.

## 3.2.1.1 Background, QA/QC, and Blank Samples and Frequency

Background samples will not be collected. Field duplicate soil samples will be collected at a rate of 1 duplicate sample per 10 field samples (10%). Duplicate samples will be analyzed for the same parameters as the field samples. Matrix spike/matrix spike duplicate, trip blank, and temperature blank samples will be not be collected for waste characterization sampling. Equipment blanks and rinsate blanks will not be collected, as WESTON will utilize disposable sampling equipment during sample collection.

#### 3.2.2 Procedures

#### 3.2.2.1 Sampling Procedures for Chemical Analyses

Demolition and oversized debris characterization samples will consist of one composite sample per debris type (concrete, asphalt, etc.) A representative sample of each debris type shall be collected and analyzed based on the physical properties of the debris type, Federal, State and local requirements, and the requirements of the accepting facility.



# 3.2.2.2 Field Quality Control Sampling Procedures

## **Equipment Blanks and Rinsate Blanks**

Equipment and rinsate blanks will not be collected. WESTON will utilize disposable sampling equipment for each sample collected to avoid the need for decontaminating sampling equipment.

#### **Duplicates**

Due to the small anticipated number of samples (i.e., less than 10), duplicates samples will not be collected for analysis. Rather, the representative samples will be of sufficient quantity to allow the laboratory to repeat any or all of the selected analyses in the event of data validity concerns.

# Matrix Spikes/Matrix Spike Duplicates (MS/MSD) Samples

Matrix spike/matrix spike duplicate, trip blank, and temperature blank samples will not be collected for characterization sampling of demolition and oversized debris.

#### 3.2.2.3 Decontamination Procedures

WESTON will use disposable sampling equipment so that decontamination will not be required. If any reusable equipment is utilized, it will be decontaminated with a non-phosphate laboratory detergent rinse (liquinox/water wash), a potable water rinse, a double deionized water rinse, and air-drying. Decontamination fluids will be containerized for testing prior to on-site treatment and disposal.

#### 3.3 CHARACTERIZATION OF DECONTAMINATION LIQUIDS

#### 3.3.1 Rationale/Sampling Locations

Decontamination liquids will be collected and containerized. Decontamination fluid will be analyzed for total Volatile Organic Compounds (VOCs). Analytical results and accompanying laboratory quality control reports will be reviewed and approved prior to discharging fluids to the Building 63 Chemical Waste Treatment Plant (CWTP).

# 3.3.1.1 Discrete/Composite Fluid Sampling Requirements

One discrete (grab) sample will be collected per 500 gallons of decontamination liquid for total VOC analysis.

# 3.3.1.2 Sample Collection and Field and Laboratory Analysis

Liquid samples will be collected by lowering the collection bottle or remote-sampling device into the container. The collection bottle will be slowly submerged to collect the sample. The sample bottles will be filled as full as possible.

# 3.3.1.3 Background, QA/QC, and Blank Samples and Frequency

Background samples will not be collected.



# 3.3.1.4 Sampling Procedures for Chemical Analyses

Fluid samples will be submitted to the contract laboratory for analysis of VOC via EPA method 8260.

# 3.3.1.5 Field Quality Control Sampling Procedures

# **Equipment Blanks and Rinsate Blanks**

Equipment and rinsate blanks will not be collected. WESTON will utilize disposable sampling equipment for each sample collected to avoid the need for decontaminating sampling equipment and collecting equipment and rinsate blanks.

# **Duplicates**

In the event that at least 10 samples of decontamination liquids are collected (i.e., greater than 5000 gallons generated), field duplicate samples will be collected at a rate of one duplicate per ten (10) field samples collected

## Matrix Spikes/Matrix Spike Duplicates (MS/MSD) Samples

Matrix spike/matrix spike duplicate, trip blank, and temperature blank samples will not be collected for characterization sampling of decontamination liquid.

#### 3.3.1.6 Decontamination Procedures

WESTON will use disposable sampling equipment so that decontamination will not be required. If any reusable equipment is utilized, it will be decontaminated with a non-phosphate laboratory detergent rinse (liquinox/water wash), a potable water rinse, a double deionized water rinse, and air-drying. Decontamination fluids will be containerized for testing prior to on-site treatment and disposal.



# 4. SAMPLE CHAIN OF CUSTODY/DOCUMENTATION

#### 4.1 FIELD DOCUMENTATION

The field documentation should enable the sampling activity to be reconstructed without relying on the collector's memory. Documentation should be kept in the field member's possession or in a secure place during fieldwork. The following topics should be recorded:

- Date and time of sampling
- Date and time of removal action
- Sample identification
- Sample location including: a hand drawn sketch of the area showing landmarks. The sample location shall be measured from a minimum two permanent of to landmarks, and shall be accurate to within 1foot.
- Depth of sample, if applicable (i.e. location in stockpile)
- Observations including descriptions of material sampled, odors, etc.
- Weather conditions at time of sampling
- Printed name of sampling personnel

#### 4.2 SAMPLE NUMBERING SYSTEM

Each sample collected will be given a unique sample designation. The sample designation will use the scheme outlined in USACE EM 200-1-3.

SAEP	01	ES	EA1	1	Comp
Sample type: ES: Excavated Soil OD: Oversized Debris M: MS/MSD		DD: Demolitio DF: Decontam: D: Duplicate			

Project Code Year Sample Type Site No. Sample No.

• For example, SAEP-01-ES-EA1-1-Comp = Stratford Army Engine Plant, 2001, Excavated Soil, Excavation Area 1, sample 1, composite.

Additional sample type/location codes may be added at the direction of the CO.



#### 4.3 SAMPLE DOCUMENTATION

## 4.3.1 Sample Labels and/or Tags

Sample labels will be consistent with the requirements of EM 200-1-3. Sample tags will not be used.

Field personnel will be responsible for identifying, labeling, providing proper preservation, and packaging samples to preclude breakage during shipment.

Every sample will be labeled and labels will include:

- Place of collection (site name).
- Unique sample number.
- Sampling date and time.
- Initials of sampling technician.
- Analysis required.
- Method of sample preservation/conditioning.
- Designation between grab and composite samples.

## 4.3.2 Sample Field Sheets and/or Logbook

The system for identifying and tracking the samples and associated field data will be recorded in a permanently bound and weatherproof notebook maintained by the field team. Team members will record all information related to sampling procedures as specified in Section 5. Field documentation will be done in indelible ink.

#### 4.3.3 Chain-of-Custody Records

Chain-of-custody records provide documentation of the handling of each sample from the time of its collection to its destruction. Sample custody will be initiated by the Contractor upon collection of samples. Chain-of-custody forms will be completed and placed in resealable waterproof plastic bags and taped to the inside lid of the cooler. The cooler will be sealed with chain-of-custody seals (a minimum of two signed custody seals on the outside with one on the front and one on the rear of the cooler covered with clear tape). Chain-of-custody forms will be used for recording pertinent information about the types and numbers of samples collected and shipped for analysis. Sample identification numbers will be included on the chain-of-custody form to ensure that no error in identification is made during shipment. Chain-of-custody procedures shall be carried out in accordance with U.S. EPA and USACE Sample Handling Protocol (Appendix F of EM 200-1-3).

## 4.4 DOCUMENTATION PROCEDURES

Prior to sample collection, labels will be affixed to sample containers. Indelible waterproof ink will be used for all logbook, chain-of-custody, and sample label entries. Documentation will conform to Appendix F of EM 200-1-3.



# 4.5 CORRECTIONS TO DOCUMENTATION

All original data recorded in field logbooks, sample labels, chain-of-custody records, and receipt for sample forms will be written in waterproof ink. If an error is made, a single line should be drawn through the entry and the entry initialed and dated. The erroneous information should not be obliterated. Any errors found in documentation should be corrected by the person who made the entry or by a designated responsible person.



# 5. SAMPLE PACKAGING AND SHIPPING

Samples will be placed in correctly labeled containers compatible with the intended analysis and properly preserved prior to shipment to the laboratory. Samples will be shipped via overnight delivery or hand-delivered to the receiving laboratory.

Each sealed container will be placed in a leakproof plastic bag. As much air as possible will be removed from the bags. Strong thermal ice chests will be filled approximately three inches with an inert protective material, such as vermiculite, bubble wrap, etc. Bagged ice/gel packs or equivalent will be placed on top of the sample containers, as necessary to ensure that the samples are cooled to at least 4° C.

This packaging and shipment will be in accordance with EPA and USACE protocol (Appendix F of EM 200-1-3). Prior to shipment, a QC check will be performed by the QC Officer to ensure samples have been properly identified and packaged, and that appropriate documentation (chain-of-custody) will accompany them.



# 6. DECONTAMINATION LIQUIDS

Decontamination liquids generated during this project (i.e., personnel and equipment decontamination) will be containerized. The decontamination liquids will be sampled for total VOCs in order to determine compliance with the 100 ppb total VOCs influent limit for the on-site Chemical Waste Treatment Plant (CWTP). The sample results will be provided to SAEP and CENAE representatives for review and approval prior to discharge of decontamination liquids to the CWTP.



# 7. QUALITY CONTROL REPORTS (DQCR)

Daily inspection/quality control reports (DQCRs) will be completed as part of the daily site inspection report. Summary reports will be submitted on a weekly basis.

Laboratory quality control information will be submitted together with the analytical data packages, as they are received from the contract laboratory (following completion of laboratory analyses). No additional quality control reports will be required.

#### 7.1 DEPARTURE FROM APPROVED PLANS

WESTON will document and report all major departures from approved plans. The report will address the following:

- Reasons for departures.
- Problems identified.
- Corrective actions.
- Effect of the departure on scope and results.

These reports of significant problems will be sent to the Contracting Officer's Representative (COR) within 48 hours of the occurrence.

#### 7.2 DATA REPORTS

The final data from the analyses will be obtained from the laboratory within 30 days of completion of laboratory work; evaluated/validated data will be reported to CENAE within 30 days of receipt of the final laboratory data package. The data shall include a table that matches primary (field) samples with their corresponding QC samples. The data reporting will also include a discussion of any problems that occurred during analyses, effects of those problems, and recommendations for further analyses, if required.



# 8. CORRECTIVE ACTIONS

#### 8.1 FIELD CORRECTIVE ACTION

The initial responsibility for monitoring the quality of field measurements and observations lies with the field personnel. The QC Officer is responsible for verifying that QC procedures are followed. This requires that the QC Officer assess the correctness of field methods and the ability to meet QA objectives. If a problem occurs that might jeopardize the integrity of the project or cause some specific QA objective not to be met, the QC Officer will notify the Project Manager. An appropriate corrective action will then be decided upon and implemented. The QC Officer will document the problem, the corrective action, and the results. Copies of the documentation will be provided to the Project Manager and Construction QC Manager.

#### 8.2 LABORATORY CORRECTIVE ACTION

The initial responsibility to monitor the quality of an analytical system lies with the analyst. The analyst will verify that all QC procedures are followed and that the results of an analysis of QC samples are within acceptance criteria. This requires that the analyst assess the correctness of all of the following items as appropriate:

- Sample preparation procedure.
- Initial calibration.
- Calibration verification.
- Method blank result.
- Laboratory control standard.
- Duplicate analysis.
- Fortified sample result.

If the assessment reveals that any of the QC acceptance criteria are not met, the analyst must immediately assess the analytical system to correct the problem. The analyst notifies the appropriate supervisor and laboratory QA coordinator of the problem and, if possible, identifies potential causes and corrective action.

The nature of the corrective action obviously depends on the nature of the problem. For example, if continuing calibration verification is determined to be "out of control," the corrective action may require re-calibration of the analytical system and re-analysis of all samples since the last acceptable continuing calibration standard.

When the appropriate corrective action measures have been defined and the analytical system is determined to be "in control," the analyst documents the problem and the corrective action. Data generated concurrently with an "out-of-control" system will be evaluated for usability in light of the nature of the deficiency. If the deficiency does not impair the usability of the results, data will be reported and the deficiency noted in the case narrative. Where sample results are impaired, the laboratory QA coordinator is notified and appropriate corrective action (e.g., re-analysis, etc.) is taken.



# 9. PROJECT SCHEDULE

The Project Schedule is presented in Figure 2-1 of the Work Plan.



# 10. SAMPLING APPARATUS AND FIELD INSTRUMENTATION

A list of the field equipment, containers, and supplies anticipated for this project is provided below.

# Field Equipment:

- Cameras
- Duct tape
- Film
- Fire extinguisher
- Garbage bags
- Indelible ink
- OVA
- MiniRAM (realtime aerosol monitor).
- Paper towels
- Razor knife
- Sample containers provided by laboratory
- Chain-of-custody forms and seals
- FSAP/CSAP
- Calibration standards
- Instrument operating manuals
- Backup field screening instruments

# **Decontamination Equipment:**

- Liquinox (nonphosphate detergent)
- Deionized water
- Decon tubs
- Scrub brushes
- Spray bottles
- Squeeze bottles
- Plastic sheeting
- DOT drums

#### Personal Protective Equipment:

- Safety goggles
- Nitrile gloves
- ANSI boots
- Hard hats
- Hearing protection
- Tyvek suits
- Level C and Level B PPE

# Sampling Equipment:

- Vermiculite or bubble wrap
- Scoops



- Scoopulas
- Stainless steel bowls
- Hand auger
- Hammer
- Shovel
- Rotary hammer drill
- 1-inch diameter carbide bits
- Aluminum pans
- Plastic scoops
- Tape measure
- Folding ruler
- Hazard shipping labels
- Cooler
- Strapping tape
- Ice
- Site plans and forms
- Rope



# **QUALITY ASSURANCE PLAN**

# **MITKEM CORPORATION**

175 Metro Center Boulevard Warwick, Rhode Island 02886-1755

Contact: Mr. Paul A. Senecal (Vice President)

**Phone**: (401) 732-3400

Fax: (401) 732-3499

# **QUALITY ASSURANCE PLAN**

Date Revised: 10/01/00

Burie D. Ruson	
QA/QC Director	Date /
Kin Chin	10/3/cN
Lab Director	Date
	QAP Control No.

Approved By:

MITKEM Corporation 175 Metro Center Boulevard, Warwick, RI 02886 Phone Number: (401) 732-3400, Fax Number: (401) 732-3499

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# 3.0 INTRODUCTION

MITKEM Corporation is a 8 (a) minority-owned environmental services company, incorporated in the State of Rhode Island.

Offices and laboratories are located in Warwick, Rhode Island. The laboratory occupies approximately 8700 square feet of laboratory space.

This Quality Assurance Plan (QAP) describes the policies, organization, objectives, quality control activities and specific quality assurance functions employed at MITKEM, and demonstrates MITKEM's dedication to the production of accurate, consistent data of known quality. This QAP is developed by following the guidelines discussed in the EPA Requirements for Quality Assurance Project Plans for Environmental Data Operations, EPA QA/R-5, Interim Final, Jan., 1994

# 4.0 QUALITY ASSURANCE POLICY STATEMENT

MITKEM is firmly committed to the production of valid data of known quality through the use of analytical measurements that are accurate, reproducible and complete. To ensure the production of such data, MITKEM has developed a comprehensive Quality Assurance/Quality Control Program that operates throughout the entire organization.

Quality Control is defined as an organized system of activities whose purpose is to demonstrate that quality data are being produced through documentation. Quality Assurance is more broadly defined as a system of activities designed to ensure that the quality control program is actually effective in producing data of the desired quality.

Quality Control is included as part of Quality Assurance. In supporting government regulatory and enforcement proceedings, a high degree of attention to quality is essential. Thorough application of quality control principles and routine quality assurance audits are required.

The basic components of the MITKEM QA/QC Program are control, evaluation and correction.

<u>Control</u> ensures the proper functioning of analytical systems through the implementation of an orderly and well-planned series of positive measures taken prior to and during the course of analysis including quality control practices, routine maintenance and calibration of instruments, and frequent validation of standards.

<u>Evaluation</u> involves the assessment of data generated during the control process. For example, precision and accuracy are determined from the results of duplicates and spikes, and other check samples. Long-term evaluation measures include performance and systems audit conducted by regulatory agencies, as well as the MITKEM quality assurance group.

Correction includes the investigation, diagnosis and resolution of any problems detected in an analytical system. Proper functioning of the system may be restored through method re-evaluation, analysis of additional check samples, trouble-shooting and repair of instrumentation or examination and comparison with historical data. Corrective actions are documented and reviewed to make sure they are implemented.

MITKEM Management considers Quality Assurance/Quality Control to be of the highest importance in the success of its Analytical Testing Laboratory and therefore fully supports the staff in the implementation and maintenance of a sound and thorough Quality Assurance Program.

# 5.0 QUALITY ASSURANCE MANAGEMENT, ORGANIZATION AND RESPONSIBILITY

Quality Assurance at MITKEM is a company-wide function that depend on:

- (1) cooperative working relationships at all levels within the laboratory and
- (2) multi level review through all working levels of responsibility.

Responsibilities for QA/QC functions begin with the bench scientist and extend to the chief executive officer.

The primary level of quality assurance resides with the bench scientist. After completion of the documented training program, his/her responsibilities include:

- complying with all aspects of formally approved analytical methods and SOPs,
- carefully documenting each step of the analytical process,
- conscientiously obtaining peer review as required,
- promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality, and
- participation in corrective actions as directed by the laboratory supervisor

The supervisor of each laboratory is responsible for ensuring thorough oversight of the quality of the data generated by the bench scientists. The laboratory supervisor implements and monitors the specific QC protocols and QA programs with the laboratory to ensure a continuous flow of data meeting all control protocols and Mitkem QA requirements. The laboratory supervisor's responsibilities include providing the bench chemist with adequate resources to achieve the desired quality of performance.

The MITKEM organizational structure is shown in the Organization Chart. Resumes of the CEO/Technical Director, Vice President, Quality Assurance Director, Operations Manager, Organic and Inorganic Managers, Chief Financial Officer, Marketing Director, Account Director, Project Managers, supervisors for the Inorganic Laboratory, GC Laboratory, Semi-volatile Organic Laboratory, Volatile Organic Laboratory and Sample Preparation Laboratory are included.

Implementation of the entire Quality Assurance Program is the responsibility of the QA Director. While interacting on a daily basis with laboratory staff members, the QA Director remains independent of the laboratories and reports directly to the Chief Executive Officer. The QA Director evaluates laboratory compliance with respect to the QA program through informal and formal systems and performance audits as described in Section 13.0. Remedial action, to alleviate any detected problems, is suggested, if necessary.

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With input from the appropriate staff members, the QA Director writes, edits and archives QA Plans, QC protocols, safety procedures, and Standard Operating Procedures (SOPs) in accordance with US EPA approved methodologies, and GLP procedures. If site specific or project specific QA Plans and/or QC protocols are needed, these will be generated as needed.

An essential element of the QA program is record keeping and archiving all information pertaining to quality assurance including QA/QC data, pre-award check sample results and scores. Performance evaluation sample results and scores, state certifications of the laboratory, external and internal audits and resolution of EPA and other audit team comments, recommendations and reports are also included. The QA Director also plays an important role in the corrective action mechanism described in Section 16.

In addition, the QA Director works with scientists and management to continually upgrade procedures and systems to improve the laboratory's efficiency and data quality.

Ultimately, the success of the QA program depends on the cooperation and support of the entire organization. The MITKEM laboratory's most valuable resource is its staff of dedicated professionals who take personal pride in the quality of their performance.

Mitkem Corporation's personnel job descriptions:

Responsibilities of each staff area in the laboratory include:

Bench Scientist / Preparation Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs
- Carefully documenting each step of the analytical process
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems
- Conscientiously obtaining peer review as required
- Promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality
- Routine housekeeping duties for their laboratory area

Bench Scientist / Instrument Laboratory Areas:

- Analysis of samples through compliance with all aspects of formally approved analytical methods and laboratory SOPs
- Routine maintenance of instrumentation
- Preparation of analytical standards and spiking solutions which are documented and traceable to their original source

- Carefully documenting each step of the analytical process
- Noting in the appropriate logbook area any unusual occurrences or sample matrix problems
- Conscientiously obtaining peer and supervisor review as required
- Promptly alerting laboratory supervisors and/or QA staff members to problems or anomalies that may adversely impact data quality
- Routine housekeeping duties for their laboratory area

# Supervisor:

- Oversight of bench scientists in their laboratory areas
- Monitors the status of all work in their laboratory area to insure compliance with holding time and turnaround time requirements
- Training new scientists in the appropriate procedures and methods in the laboratory
- Works with laboratory managers and the QA staff to review, revise and implement SOPs
- Insures adequate resources to perform the needed tasks by working with administrative personnel to order needed supplies
- Insures all supplies and reagents meet the QC requirements of their intended task prior to their use in the laboratory
- Insures all staff are using proper safety protocols
- Works with laboratory managers on the annual review of personnel performance
- Interviews prospective new employees to insure they have the minimal level of qualifications, experience, education and skills necessary to perform their tasks, as well as the appropriate work ethic and social skills necessary for proper teamwork and productivity

#### Senior Scientists

- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client
- Documents any non-compliance or other unusual occurrences noted during sample analysis
  and data review such that these can be included in the report narrative and explained to the
  client
- Assist laboratory Managers and Supervisors in other tasks as required

#### Laboratory Managers

- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client
- Oversight of Supervisors and Senior Scientists in their laboratory areas
- Assists analysts and Supervisors in the troubleshooting and maintenance of instrumentation

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- Works with instrument suppliers to insure appropriate instruments are available in the laboratory
- Provides technical assistance to other laboratory staff

# **Operations Manager**

- Prioritizes work in the laboratory areas to insure projects are completed on a timely basis
- Works with laboratory Managers and Supervisors to coordinate laboratory areas in the completion of analytical projects
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client
- Writes project report narratives to document any unusual occurrences noted during sample analysis
- Works with management and supervisory staff to continuously improve the quality and efficiency of all company procedures
- · Works with clients to insure all questions and concerns are addressed and answered
- · Assists laboratory Managers and Supervisors in the annual review of personnel performance
- Supervises laboratory Managers and Supervisors to insure compliance with company QA policies and other company procedures

# Project Manager

- Works with the client to completely understand the requirements of all incoming work
- To evaluate the client's requirements as compared to the abilities of the laboratory
- To communicate the customer's requirements to all laboratory staff working on the project
- Works with the customer to determine the number and type of sample containers required for the project
- Works with the Sample Custodian to resolve and communicate to the client any problem or discrepancies with incoming samples
- Maintains open, responsive and continuous communication with the customer.
- Follows up with the client to assess level of satisfaction, and insure all project goals have been accomplished.

# QA Manager

- Implements the entire QA program
- Interact on a daily basis with laboratory staff
- Evaluates compliance with the QA program through formal and informal reviews of data and processes
- Implements the corrective action system

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- Works with laboratory Managers and Supervisors to implement new SOPs and to annually review and revise existing SOPs
- Interfaces with certification authorities and agencies to maintain existing certifications and obtain new certifications
- Maintains records of employee training and certification

# **Technical Director**

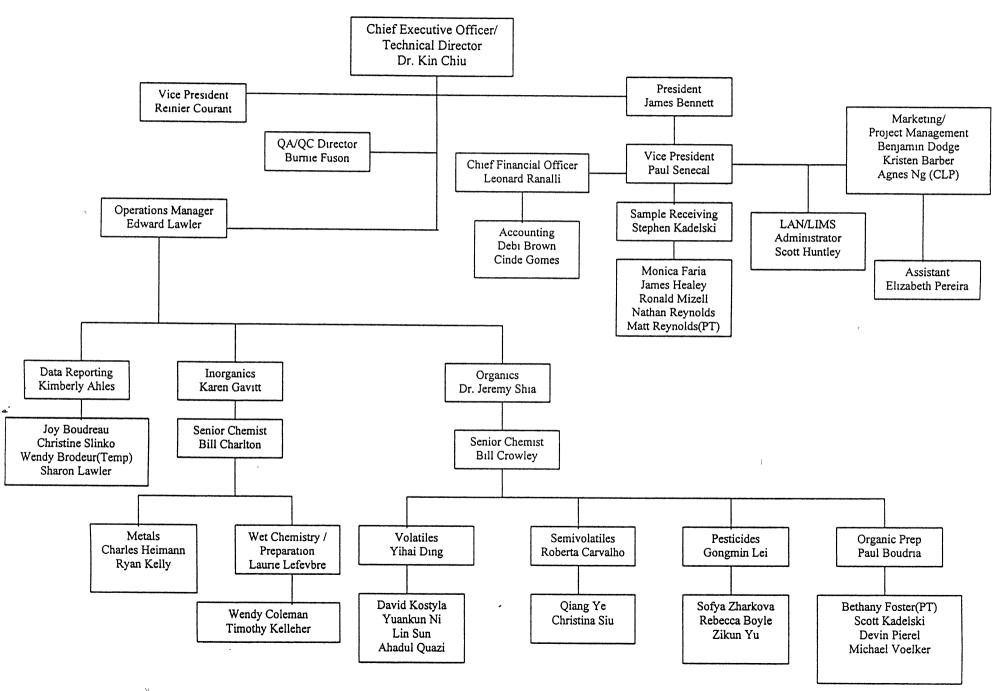
- Review of analytical data to insure compliance with method/SOP requirements prior to release to the client
- Supervises all Management, QA and Supervisory staff to insure compliance with company QA policies and other company procedures
- Provides technical assistance to all areas of the laboratory staff
- Works with clients to insure their understanding of complex technical issues

The personnel training records are located in the QA department. All individual training is documented including new employee training, individual training, and Health and Safety training.

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# Figure 5-1 MITKEM Corporation's Organizational Chart

Figure 5-1
MITKEM Corporation's Organizational Chart
Revised 08/01/00



### KIN S. CHIU

# Chief Executive Officer/Technical Director

Dr. Chiu is a MIT trained mass spectroscopist with extensive experience in the trace level analyses of organic and hazardous waste compounds in environmental samples. He has over twenty years of experience in using GC/MS, HPLC and GC with various detectors. He has been involved with research and development on non-routine analytical approaches to environmental chemistry problems. Dr. Chiu has been the lead chemist responsible for analytical laboratory operations at several of the most respected laboratory facilities in the northeast.

Dr. Chiu has extensive program management experience through positions of high responsibility in Contract Laboratory Program (CLP) laboratories. He also has significant experience with the management of programs involving Army Corps of Engineers, Navy and Air Force analytical requirements.

Dr. Chiu also has extensive experience with the financial and business management responsibilities of small and medium size corporations, as well as the public sector. MITKEM is his second environmental laboratory start-up. The first, CEIMIC Corporation was also highly successful. He was an active partner in both the technical and business aspects of both companies.

**EDUCATION** 

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

Cambridge, Massachusetts

Chemistry, PhD

**RUTGERS UNIVERSITY** 

New Brunswick, New Jersey Environmental Sciences, MS

UNIVERSITY OF MARYLAND

College Park, Maryland Chemistry, BS

# RELATED EXPERIENCE

1994-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Chief Executive Officer
- Technical Director

1993

COAST TO COAST ANALYTICAL

Westbrook, Maine

- Director of Laboratory Operations

1991-1993

MASSACHUSETTS WATER RESOURCES

**AUTHORITY** 

Boston, Massachusetts

- Laboratory Superintendent

1988-1992

CEIMIC CORPORATION

Narragansett, Rhode Island

- Vice President Organic Laboratory Operations and

Technical Director

1983-1988

**ENSECO/ERCO DIVISION** 

Cambridge, Massachusetts

- Head of Research and Development

#### REINIER A. COURANT

#### Vice President

Mr. Courant has over twenty five years of experience in environmental chemistry. He has managed a number of large scale multi-disciplinary and international environmental baseline studies. These studies involved the collection and analysis of samples for a wide variety of parameters, evaluation and interpretation of the generated data, and writing of the final report. Mr. Courant has authored 25 scientific papers, taught chemistry at the university level and held senior scientist and project manager positions as well as upper management and partner positions in several environmental firms.

Mr. Courant has extensive experience in many phases of environmental chemistry, with particular concentration in laboratory design and automation, specifically in electronic transfer of data and set-up of information management systems. Mr. Courant also has considerable experience in sample analysis, data review and data package preparation for EPA Contract Laboratory Program inorganic sample analyses. Mr. Courant's experience with chemical analysis instrumentation is wide-ranging, with a primary focus on elemental and trace metals analyses.

In the past ten years he has been involved in the start-up and senior management of several environmental testing laboratories.

**EDUCATION** 

UNIVERSITY OF RHODE ISLAND

Graduate School of Oceanography Kingston, Rhode Island

Chemical Oceanography, MS

NORTHEASTERN UNIVERSITY

Boston, Massachusetts Mathematics, MS

DELFT INSTITUTE OF TECHNOLOGY

Delft, Netherlands

Chemistry

### RELATED EXPERIENCE

1994-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Vice President

1991-1994	CC CORPORATION Lexington, Massachusetts - President
1987-1991	CEIMIC CORPORATION Narragansett, Rhode Island - Vice President
1985-1987 1980-1983	ENERGY AND ENVIRONMENTAL ENGINEERING, INC. Cambridge, Massachusetts - Vice President
1983-1985	RESEARCH PLANNING INSTITUTE Columbia, South Carolina - Senior Chemist Niger Delta Baseline Studies
1978-1980	INTERSTATE ELECTRONICS CORPORATION Anaheim, California - Senior Oceanographer US EPA Studies of US Offshore Dumpsites
1976-1978	ENERGY RESOURCES COMPANY - ERCO Cambridge, Massachusetts - Field Operation Manager and Senior Oceanographer Georges Bank Region Environmental Baseline Studies
1972-1976	UNIVERSITY OF RHODE ISLAND Kingston, Rhode Island - Research Specialist/Graduate Student
1969-1972	WOODS HOLE OCEANOGRAPHIC INSTITUTE Woods Hole, Massachusetts - Research Assistant/Graduate Student

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# Burnie D. Fuson Quality Assurance Director

Mr. Fuson has sixteen years of experience in environmental chemistry. He has been the laboratory technical consultant for many clients including the U.S. Army Corps of Engineers, Air Force, Navy, Department of Defense, Department of Energy, and EPA Regions I, IV, V, and IX. He has also been advisor to the laboratory Director for special technical/analytical functions. His knowledge and experience encompasses all analytical aspects of an environmental laboratory including GC/MS (Volatiles and Semi-volatiles), GC (Pesticides, Herbicides, PCBs, PAHs), HPLC (PAHs and Explosives), IC, ICP, AA, TOX, TOC, Inorganic and Organic sample preparation, general chemistry, and Data validation.

Mr. Fuson also has eight years experience as Quality Assurance Officer. His responsibilities included acquisition and renewal of government, state, and local certifications. The establishment and updating of the laboratory QA Manual, Standard Operating Procedures, laboratory documentation (such as logbooks), accuracy and precision statistical data, and QA reports to management. Responsibilities also included monitoring analytical functions with respect to EPA and state protocols, issue recommendations and corrective action for laboratory QA/QC requirements, perform internal and external audits, and provide and evaluate blind QC check samples. He established new techniques and programs for advanced analytical services including low level cyanide analysis for samples with high sulfur concentration matrices, explosives analysis using HPLC and IC, Solid Phase extraction of TRPH, Pesticide, PAH, and semi-volatile samples, and mass spectral identification of C-S ring systems in high pH matrices.

As Health and Safety Officer, he was responsible for all health and safety aspects of the laboratory including OSHA protocols established in 29 CFR 1900.1000, waste disposal and recovery, chemical exposure limits for laboratory personnel, and emergency evacuation systems.

**EDUCATION** 

UNIVERSITY OF WEST FLORIDA

Pensacola, Florida Chemistry, BS

RELATED EXPERIENCE

1999-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Ouality Assurance Director

1990-1999

LAW ENG. & ENVIRON. SVCS.

Pensacola, Florida

- Technical Coordinator
- Quality Assurance Officer
- Operations Manager
- Health & Safety Officer

1989-1990

ENVIRON. CONTROL TECH, INC.

Ann Arbor, Michigan

- Senior Project Chemist

1984-1986

PIONEER LABORATORY, INC.

Pensacola, Florida

- GC/MS Chemist

#### EDWARD A. LAWLER

# Operations Manager \*

Mr. Lawler has over twenty years of experience in environmental laboratory operations. He has extensive experience in managing laboratory workflow and in establishing and maintaining customer relationships. He also has considerable experience in a wide range of environmental chemical analyses, with a concentration in trace level volatile organics analysis.

Mr. Lawler's responsibilities include coordination and prioritization of all analytical chemistry testing at Mitkem. This includes daily meetings with the organic and inorganic laboratory supervisors and managers to insure all technical and schedule requirements are met. Mr. Lawler also reviews laboratory data to insure QA/QC criteria have been achieved, and provides final review of data reports prior to delivery to clients. Mr. Lawler also manages certain significant analytical testing programs, acting as principal technical liaison with the client.

Mr. Lawler's previous experience includes various staff, management and senior management positions at several environmental testing laboratories. Direct project experience includes EPA CLP, Army MRD, Navy NEESA and NFESC, DOD HAZWRAP and New York DEC ASP programs. Mr. Lawler has also provided expert testimony at several Superfund trials including pre-trial consulting and trial witness services.

**EDUCATION:** 

UNIVERSITY OF MASSACHUSETTS

Amherst, Massachusetts Environmental Sciences, BS

### **RELATED EXPERIENCE:**

1997-Present

MITKEM CORPORATION

Warwick, Rhode Island -Operations Manager

1989-1997

NATIONAL ENVIRONMENTAL TESTING,

CAMBRIDGE DIVISION

Bedford, Massachusetts

-Division Manager

-Proposal/Contract Manager

-Director of Project Management

1983-1989

CAMBRIDGE ANALYTICAL ASSOCIATES, INC.

Boston, Massachusetts

-Project Manager

-Volatile Organic Laboratory Manager

1978-1983

ENERGY RESOURCES COMPANY, INC. - ERCO

Cambridge, Massachusetts

-Volatile Organics (GC) Manager

-Analytical Chemist-Volatile Organics Lab

-Analytical Chemist-Organic Preparation Lab

1978

LAPUCK LABORATORIES, INC.

Watertown, Massachusetts

-Analytical Chemist-Wet Chemistry & Metals

-Microbiologist

# CHIH-PING (JEREMY) SHIA

# Organic Laboratory Manager

Dr. Shia is an analytical organic chemist with extensive experience in the analysis of environmental samples using various spectroscopic techniques. He has performed a wide variety of both routine and research and development analytical testing, including the implementation of new state of the art methodology.

Dr. Shia's responsibilities include providing technical chemistry support to the Supervisors and staff of Mitkem's volatiles, semivolatiles, gas chromatography and sample preparation laboratories. He actively coordinates technical projects to insure all QA/QC requirements are met, including both routine and special project requirements. Dr. Shia plays a leading role in the development and implementation of organic laboratory Standard Operating Procedures, and oversees statistical instrument and method performance studies. Previously Dr. Shia worked in chemistry research and teaching roles at two universities.

**EDUCATION** 

UNIVERSITY OF MASSACHUSETTS

Lowell MA Chemistry, Ph.D.

NATIONAL TAIWAN UNIVERSITY

Taipei, Taiwan Chemistry, BS

# RELATED EXPERIENCE

1996-Present

### MITKEM CORPORATION

Warwick, Rhode Island

Organic Laboratory Manager

Volatile Organic Laboratory Supervisor

- Senior Chemist

1990-1994

# UNIVERSITY OF MASSACHUSETTS

Lowell, MA

- Teaching Assistant

1988-1989

### NATIONAL TAIWAN UNIVERSITY

Taipei, Taiwan

- Research Assistant

# YIHAI DING

# Chemist, GC/MS and GC Laboratories

Mr. Ding has experience in a wide variety of analytical chemistry techniques, including GC, GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation.

Mr. Ding's responsibilities include the daily tuning and calibration of analysis instrumentation, monitoring QC criteria, sample analysis, review of results and mass spectra data, use of CLP forms generation software and other statistical data evaluation software.

Mr. Ding's prior experience includes research into the mechanisms and kinetics of various biochemical processes. A large portion of this research has required the analysis of reactants and products using state-of-the-art chemical instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level.

# **EDUCATION**

# MIDDLE TENNESSEE STATE UNIVERSITY

Murfreesbro, Tennessee - Chemistry, MS

### JILIN UNIVERSITY

Changchun, China

- Biochemistry, BS

#### RELATED EXPERIENCE

1998-Present

# MITKEM CORPORATION

Warwick, Rhode Island

- GC/MS Analyst

1994-1998

# MIDDLE TENNESSEE STATE UNIVERSITY

Murfreesbro, Tennessee

Researcher

- Laboratory Instructor, chemistry and biochemistry

1993-1994

# NATIONAL ENZYME ENGINEERING LAB

Changchun, China

- Researcher

#### KAREN M. GAVITT

# Inorganic Laboratory Manager

Ms. Gavitt has nearly ten years of experience in the analysis of environmental and hazardous waste samples for both organic and inorganic analytes. A large portion of this experience has involved the use of axial ICP, radial ICP, cold vapor AA and graphite furnace AA for trace metals analysis.

Ms. Gavitt's responsibilities at Mitkem include management of the inorganic chemistry laboratories including metals and conventional wet chemical analyses. Her duties include the day-to-day scheduling of all analytical work in her department to meet program turnaround and method holding time requirements. Ms. Gavitt is also responsible for the technical and QC performance of a wide variety of methods, as well as development and implementation of Standard Operating Procedures, method and instrument performance measures, daily review of sample and QC data, training of laboratory staff and discussion of program requirements and project status with Mitkem's project managers and clients.

She was a GC/MS analyst during her most recent employment before joining Mitkem. Ms. Gavitt also has experience in the analysis of samples for inorganic and organic analyses by US EPA CLP protocols, New York State ASP protocols and various DOD analytical programs.

**EDUCATION** 

DUQUESNE UNIVERSITY

Pittsburgh, Pennsylvania

Chemistry, BS

# RELATED EXPERIENCE

1994-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Inorganic Laboratory Manager

1994

**ENVIRONMENTAL SCIENCES SERVICES** 

Providence, Rhode Island

- GC/MS Analyst

1990-1994

**CEIMIC CORPORATION** 

Narragansett, Rhode Island

- Trace Metals Laboratory Supervisor

- Organic Prep Lab Technician

# ROBERTA A. CARVALHO

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# Semivolatile Organic Laboratory Supervisor

Ms. Carvalho has experience with analysis of environmental samples by a variety of analytical techniques. Her responsibilities at Mitkem involve the coordination of semivolatile organics analyses using GC/MS instrumentation. Her duties in this role include instrument calibration and maintenance, sample analysis, review of sample and QC data, implementation of Standard Operating Procedures, documentation of instrument and method QC criteria and coordination with other laboratory sections.

Ms. Carvalho's experience includes GC/MS, ICP/MS, GC, IC and AA techniques. She has used this equipment in routine analysis and research roles. She has also taught chemical instrumentation courses in a university setting. Ms. Carvalho has experience with the analysis of a wide variety of terrestrial and marine sample matrices, as well as atmospheric samples. She has participated in 20 offshore research cruises involving various responsibilities including cochief scientist. Her knowledge of chemical analysis of environmental media is wide-ranging and diverse.

**EDUCATION** 

TEXAS A&M UNIVERSITY

College Station, Texas

- Chemical Oceanography, MS

TEXAS A&M UNIVERSITY

Galveston, Texas

Marine Biology, BS

### RELATED EXPERIENCE

1998-Present

MITKEM CORPORATION

Warwick, Rhode Island

- GC/MS Analyst

1998

LAWLER, MATUSKY & SKELLY ENGINEERS

Somerset, Massachusetts

Environmental Field Technican

1997

# U.S. GEOLOGICAL SURVEY

Woods Hole, Massachusetts

- Technical Consultant, Marine Geochemistry Division

1990 - 1997

# **TEXAS A&M UNIVERSITY**

College Station, Texas

- Graduate Student Teacher, Analytical Instrumentation
- Researcher, Oceanographic and Environmental Research Laboratory

# YIHAI DING

# Chemist, GC/MS and GC Laboratories

Mr. Ding has experience in a wide variety of analytical chemistry techniques, including GC. GC/MS, HPLC and FTIR. His expertise includes the operation, calibration and maintenance of sophisticated, computer controlled instrumentation.

Mr. Ding's responsibilities include the daily tuning and calibration of analysis instrumentation, monitoring QC criteria, sample analysis, review of results and mass spectra data, use of CLP forms generation software and other statistical data evaluation software.

Mr. Ding's prior experience includes research into the mechanisms and kinetics of various biochemical processes. A large portion of this research has required the analysis of reactants and products using state-of-the-art chemical instrumentation. Mr. Ding has also taught chemistry and biochemistry laboratory courses at the university level.

### **EDUCATION**

# MIDDLE TENNESSEE STATE UNIVERSITY

Murfreesbro, Tennessee Chemistry, MS

# JILIN UNIVERSITY Changchun, China

Biochemistry, BS

#### RELATED EXPERIENCE

1998-Present

MITKEM CORPORATION

Warwick, Rhode Island GC/MS Analyst

1994-1998

MIDDLE TENNESSEE STATE UNIVERSITY

Murfreesbro, Tennessee

Researcher

Laboratory Instructor, chemistry and biochemistry

1993-1994

NATIONAL ENZYME ENGINEERING LAB

Changchun, China

Researcher

# **GONGMIN LEI**

# Gas Chromatography Lab Manager

Mr. Lei has several years of experience in the analyses of samples for Pesticide and PCBs. He also has experience in the extraction of analyses of hydrocarbons in water and sediments by GC-FID and GC-MS techniques.

As manager of Mitkem's gas chromatography laboratory Mr. Lei is responsible for the daily scheduling of all GC analyses including pesticides, PCBs, herbicides, petroleum hydrocarbons, and specialty testing. Mr. Lei is also responsible for the implementation of Standard Operating Procedures, the production and review of sample and QC results, the documentation of instrument and method performance data and the training of analytical staff. Mr. Lei is familiar with the use of the Target software package to provide EPA CLP and New York State ASP deliverables on a wide variety of GC analysis methods.

Prior to joining Mitkem Mr. Lei gained extensive experience in the application of analytical chemistry techniques to a wide variety of environmental problems, both in the US and in China. He has worked as a researcher in both university and government organizations dedicated to managing environmental issues.

**EDUCATION** 

# UNIVERSITY OF MASSACHUSETTS

Boston, Massachusetts

- Environmental Sciences, MS

# NANKAI UNIVERSITY

Tianjin, China

- Environmental Analytical Chemistry, MS
- Chemistry, BS

### RELATED EXPERIENCE

1996-Present

# MITKEM CORPORATION

Warwick, Rhode Island

- Pesticide/PCB Lab Manager

1993-1996

# UNIVERSITY OF MASSACHUSETTS

Boston, Massachusetts

- Research and Teaching Assistant

1987-1993

# TIANJIN INSTITUTE OF ENVIR. PROTECTION

Tianjin, China
- Environmental Engineer

1984-1987

# NANKAI UNIVERSITY

Tianjin, China
- Research Assistant

### PAUL T. BOUDRIA

# Organic Preparation Laboratory Supervisor

Mr. Boudria is responsible for the daily workflow management and supervision of the organic sample preparation laboratory. In this role he evaluates incoming sample analysis requests, schedules sample and QC analyses, reviews data, interfaces with the supervisors of the GC and GC/MS laboratories to insure all technical and schedule requirements are met. He also provides training to laboratory staff, develops and reviews Standard Operating Procedures, implements new methods, performs and evaluates method performance documentation.

Mr. Boudria is thoroughly familiar with U.S. EPA and SW846 methodologies and sample extraction and cleanup protocols. He has also worked on a contract basis for a major pharmaceutical company specializing in hazardous and other chemical waste management, including OSHA hazardous waste operators duties and training.

**EDUCATION** 

BRIDGEWATER STATE COLLEGE

Bridgewater, Massachusetts Chemistry/Geology, BS

### RELATED WORK EXPERIENCE

1998-Present

MITKEM CORPORATION

Warwick, Rhode Island

Preparation Laboratory Supervisor

1998

PFIZER PHARMACEUTICALS

Groton, Connecticut

- Contract Hazardous Waste Specialist/Chemist

1996-1998

MITKEM CORPORATION

Warwick, Rhode Island

- Preparation Laboratory Chemist

# LEONARD A. RANALLI

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#### Chief Financial Officer

Mr. Ranalli has an extensive financial and business background. He brings to the Mitkem Corporation 18 years of banking experience. His expertise is in operations and financial management.

**EDUCATION:** 

**BROWN UNIVERSITY** 

Providence, Rhode Island

Sociology, BA

**RELATED EXPERIENCE:** 

1994-Present

MITKEM CORPORATION

Warwick, Rhode Island - Chief Financial Officer

1992-1994

OLD STONE BANK

Providence, Rhode Island - Assistant Vice President/

Commercial Real Estate Officer

1990-1992

EASTLAND BANK

Woonsocket, Rhode Island
- Assistant Vice President/
Commercial Loan Officer

1981-1990

RHODE ISLAND HOSPITAL TRUST

NATIONAL BANK

Providence, Rhode Island

- Loan Officer

- Credit Analyst

- Operations Manager, Wire Transfer Department

- Operations Manager, Purchasing Department

# PAUL A. SENECAL

# **Marketing Director**

Mr. Senecal has several years of experience in marketing and client services in the environmental laboratory industry as well as a strong scientific background. His duties include business development, project management and building and maintaining client relationships.

Mr. Senecal works with engineers, consultants and government clients to develop and define the scope of analytical chemistry programs. He has experience in the set-up and management of a wide variety of site assessment and monitoring projects. This experience includes programs performed under the auspices of the New York State DEC, the US EPA, Army and Air Force environmental agencies. He also has managed a number of large-scale analysis programs for commercial and industrial clients. He is familiar with the method and QC requirements of these analytical programs, the evaluation of samples received at the laboratory for compliance with program requirements, the communication of any technical or schedule issues developed during the sample analysis process.

Prior to his employment at Mitkem Mr. Senecal worked for a large multi-location environmental testing laboratory participating in a wide variety of government and private chemistry programs.

**EDUCATION:** 

ST. LAWRENCE UNIVERSITY

Canton, New York

Biology, BS

### **RELATED EXPERIENCE:**

1995-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Account Executive

1993-1995

PACE, INCORPORATED

Minneapolis, Minnesota

- Account Executive

- Client Services Technician

- Chemist

1992

MINNESOTA PUBLIC LOBBY

Minneapolis, Minnesota

- Field Manager

# 6.2 Representativeness:

Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. In most cases, representativeness is achieved by mixing the laboratory sample well before removing a portion for analysis. On occasion, multi-phase laboratory samples may require that each phase be analyzed individually and reported in relation to its proportion in the whole sample.

# 6.3 Completeness:

The completeness goal is 100% in all cases and includes:

- Analysis of all samples;
- Generation and analysis of all required QC samples;
- Sufficient documentation of associated calibration, tuning and standardization:
- Records of data reduction processes, including manual calculations.

While the laboratory staff is responsible for achieving the completeness objective stated above, completeness is ensured by assigning each project a specific project manager whose functions include sample management and tracking.

# 6.4 Comparability:

To assure comparability, MITKEM employs established and approved analytical methods (e.g. USEPA protocols), consistent analytical bases (dry weight, volume, etc.) and consistent reporting units (mg/Kg,  $\mu$ g/L, etc.). Where data from different samples must be comparable, the same sample preparation and analysis protocols are used for all of the samples of interest.

#### BENJAMIN F. DODGE

# **Account Executive**

Mr. Dodge oversees day-to-day program management of in-house projects and serves as technical liaison to clients. In this role Mr. Dodge is responsible for defining project scope through discussion with the client, determination of proper analytical methodology, development of price quotations, discussion of technical and schedule issues with laboratory personnel, reviewing client requests on chain of custody and sample transmittal documentation, resolution of any problems in sample delivery or documentation, review of project log-in information, monitoring project status and communication of status information to the client, discussion of results and communication of questions or technical interpretation with the client, and follow-up on completed projects.

Mr. Dodge has managed a wide variety of analytical services projects at Mitkem, including site investigation, remedial support, long-term landfill monitoring, industrial wastewater and hazardous waste programs. A number of these programs have involved the production of EPA Contract Laboratory Program (CLP) data deliverables, or New York State Analytical Services Protocol (ASP) deliverables and methodology. A significant portion of the programs managed by Mr. Dodge have involved rapid turnaround analytical services, requiring a high level of program management.

**EDUCATION** 

EASTERN CONNECTICUT COLLEGE

Willimantic, Connecticut Environmental Science, BS

### RELATED WORK EXPERIENCE

1996-Present

MITKEM CORPORATION

Warwick, Rhode Island

- Project Coordinator
- Sample Custodian

# 6.0 QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS AND COMPARABILITY

As part of the evaluation component of the overall QA Program, laboratory results are compared with the data quality indicators defined as follows:

- Precision: the agreement of reproducibility among individual measurements of the same property, usually made under identical conditions.
- Accuracy: the degree of agreement of a measurement with the true or accepted value.
- Representativeness: the degree to which data accurately and precisely represent a characteristic of a population, parameter variations of a sample of a finite process condition, or of a finite environmental condition.
- Completeness: a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions.
- Comparability: an expression of the confidence with which one laboratory data set can be compared with another laboratory data set in regard to the same property and laboratory sample population.

Quality Assurance objectives may vary by project and requested parameters. The accuracy, precision, and representativeness of data will be functions of the origins of the sample material, the procedures used to analyze samples and generate data, and the specific sample matrices involved in each project. Quality control practices utilized in the evaluation of these data quality indicators include blanks, replicates, spikes, standards, check samples, calibrations and surrogates. The process for quantifying or assessing the above indicators for data quality are addressed in Section 15.

# 6.1 Precision and Accuracy:

For each parameter analyzed, the QA objectives for precision and accuracy will be determined from:

- Published historical data:
- Method validation studies;
- MITKEM experience with similar samples and/or;
- Project-specific requirements, such as those stipulated by the USEPA in the CLP protocols and control documents.

# 7.0 SAMPLING PROCEDURES

For most projects, outside sampling teams deliver or send samples to the MITKEM laboratory. When sampling by MITKEM personnel is required, the sampling team follows the sampling procedures outlined in the EPA/SOW *Test Methods for Evaluating Solid Wastes*, SW-846, 3<sup>rd</sup> Edition, or procedures found in the EPA "Handbook for Sampling and Sample Preservation of Water and Wastewater".

Appropriately prepared sample containers are supplied by MITKEM at clients' request. When required, preservatives are added to the sample containers. Tables 7-1 through 7-3 provide the MITKEM Recommended Methods for Sampling, Sample Volume and Preservation of Samples for Analysis. Additional sample volumes may be required if additional QC functions are to be performed.

Holding times for SW846, CLP Methods, Standard Methods and certain USEPA methods are different and are presented in Tables 7-1 to 7-3.

Recommended Container, Preservation Techniques and Holding Times for SW-846 Analyses

Table 7-1

Analyte Volatile	es Organics	Method	Containers	Required* Volume	Preservation	Holding <u>Times</u>		
	Solid	8260B, 5030B	Amber glass jar with Teflon lining	Minimal head- space in jar	4°C	14 days		
	Solid <sup>a</sup>	8260B, 5035	40mL vial or Encore with Teflon lining	5.0gram ± 0.5	4°C, unpreserve	d 48 hours		
					DI Water -10 to -20°C	14 days		
					Sodium bisulfate -10 to -20°C, 4°C			
					Methanol 4 <sup>0</sup> C	14days		
	Aqueous	8260B, 5030B	40mL VOA Vials with Teflon septum	40mL	4°C HCl, pH<2	14 days		
Semivo	latile Organics	•				•		
	Solid	3540C, 3550B 8270C	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days		
	Aqueous	3510C, 3520C 8270C	Amber glass bottles with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days		
Polychl	orinated Bıphenyl	s						
	Solid	3540C, 3550B 8082	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days		
	Aqueous	3510C, 3520C 8082	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days		
Organo	chlorine Pesticide	S						
S	Solid	3540C, 3550B 8081A	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days		
	Aqueous	3510C, 3520C 8081A	Amber glass bottle with Teflon lining	IL	4°C	Extraction within 7 days Analysis within 40 days		
Chlorin	Chlorinated Herbicides							
	Solid	8151A 8151A	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days		
	Aqueous	8151A	Amber glass bottle	1L	4°C	Extraction within 7 days		

QA Plan Section 7 rev 2 Date Initiated 1/15/94 Date Revised. 07/31/00 Page 3 of 10 Analysis within 40 days

8151A with Teflon lining

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# Table 7-1 (cont'd)

# Recommended Containers, Preservation Techniques and Holding Times for SW846 Analyses

Analytes Total Petroleum H		Containers	Required* Volume	Preservation	Holding Times		
Gasoline Range Oi Solid	8015M, 5030B	Amber glass jar With Teflon lining	Minimal head- space in jar	4°C	14 days		
Solid <sup>a</sup>	8015M, 5035	40mL vial or Encore with Teflon lining	5 0gram ± 0 5	4°C, unpreserve	ed 48 hours		
				DI Water -10 to -20°C	14 days		
				Sodium bisulfat $-10$ to $-20^{\circ}$ C, $4^{\circ}$			
				Methanol 4 <sup>0</sup> C	14days		
Aqueous	8015M, 5030B	40mL VOA vials With Teflon septum	40mL	4°C HCl, pH<2	14 days		
Diesel Range Orga	nics						
Solid	3540C, 3550B 8015M	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 14 days Analysis within 40 days		
Aqueous	3510C, 3520C 8015M	Amber glass bottle with Teflon lining	1L	4°C H₂SO₄, pH<2	Extraction within 7 days Analysis within 40 days		
Total Metals excer	ot Mercury and Chromic	ım (VI)					
Solid	3050B 6010B	Amber glass jar with Teflon lining	10g	4°C	180 days		
Aqueous	3005A, 3010A	Polyethylene bottle	100mL	HNO <sub>3</sub> , pH<2	180 days		
Chromium (VI) Solid	7196A	Amber glass jar with Teflon lining	10g	4°C	Digestion within 30 days Analysis within 96 hours		
Aqueous	7196A	Polyethylene bottle	25mL	4°C	24 hours		
Mercury	Mercury						
Solid	7471A	Amber glass jar	10g	4°C	28 days		
Aqueous	7470A	Polyethylene bottle	100mL	4°C	28 days		

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Cyanide						
j	Solid	9012	Amber glass jar with Teflon lining	lg	4°C	14 days
	Aqueous	9012	Polyethylene bottle	50mL	4°C NaOH, pH>12	12 days
Flashpoi	nt Aqueous	1010	Amber glass bottle	30mL	4°C	28 days

Table 7-2

Recommended Container, Preservation Techniques and Holding Times
For
CLP/ASP Analyses

Analyt	es	Method	Containers	Required* Volume	Preservation	Holding Times	
Volatil	e Organics				1100011441011	Times	
	Solid	CLP/ASP	Amber glass jar with Teflon lining	Minimal head- space in jar	4°C	10 days from VTSR	
	Aqueous	CLP/ASP	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR	
		CLP Low	40mL VOA vials with Teflon septum	40mL	4°C HCl, pH<2	10 days from VTSR	
Semivo	olatile Organics						
	Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis within 40 days	
	Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	IL	4°C	5 days from VTSR Analysis within 40 days	
		CLP Low	Amber glass bottle with Teflon lining	lL	4°C	5 days from VTSR Analysis within 40 days	
Organo	chlorine Pesticide	/PCB					
J	Solid	CLP/ASP	Amber glass jar with Teflon lining	30gram	4°C	10 days from VTSR Analysis with 40 days	
	Aqueous	CLP/ASP	Amber glass bottle with Teflon lining	1L	4°C	5 days from VTSR Analysis within 40 days	
		CLP Low	Amber glass bottle with Teflon lining	lL	4°C	5 days from VTSR Analysis within 40 days	
Cyanide							
- <b>,</b>	Solıd	CLP/ASP	Amber glass jar	2gram	4°C	12 days from VTSR	
	Aqueous	CLP/ASP	Polyethylene bottle	50mL	4°C NaOH, pH>12	12 days from VTSR	
Total Metals except Mercury							
	Solid	CLP/ASP	Amber glass jar	2gram	4°C	180 days from VTSR	
	Aqueous	CLP/ASP	Polyethylene bottle	100mL	HNO <sub>3</sub> , pH<2	180 days from VTSR	

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# Table 7-2 (con't)

# Recommended Container, Preservation Techniques and Holding Times For CLP/ASP Analyses

Analytes Mercury	Method	Containers	Required* Volume	Preservation	Holding <u>Times</u>
Solid	CLP/ASP	Amber glass jar	10gram	4°C	26 days from VTSR
Aqueous	CLP/ASP	Polyethylene bottle	100mL	4°C HNO <sub>3</sub> , pH<2	26 days from VTSR

Table 7-3

Recommended Containers, Preservation Techniques and Holding Times for Other Analyses

Analyt Volatil	<u>es</u> e Organics	Method	Containers	Required* Volume	Preservation	Holding Times			
	Aqueous	624	40mL VOA vials	40mL	4°C	14 days			
		524.2	with Teflon septum 40mL VOA vials with Teflon lining	40mL	HCl, pH<2 4°C HCl, pH<2	14 days			
Semivolatile Organics									
	Aqueous	3510C, 3520C 625	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days			
Organo	chlorine Pesticide	PCB							
	Aqueous	3510C, 3520C 608	Amber glass bottle with Teflon lining	1L	4°C	Extraction within 7 days Analysis within 40 days			
EDB/D	ВСР								
	Aqueous	504.1	40mL VOA vials with Teflon septum	35mL	4°C HCl, pH<2	28 days			
MA Ex	tractable Petroleu	m Hydrocarbons (	ЕРН)	•		•			
	Solid	3540C, 3550B MADEP	Amber glass jar with Teflon lining	30gram	4°C	Extraction within 7 days Analysis within 40 days			
	Aqueous	3510C, 3520C MADEP	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	Extraction within 14 days Analysis withing 40 days			
MA Vo	olatile Petroleum F	Ivdrocarbons (VP)	H)						
	Solid	MADEP	Amber glass jar with Teflon lining	30gram	4°C 15mL Methanol	14 days			
	Aqueous	MADEP	40mL VOA vial with Teflon lining	40mL	4°C HCl, pH<2	14 days			
Oıl & C	rease				1				
	Aqueous	1664	Amber glass bottle with Teflon lining	1L	4°C HCl, pH<2	28 days			
Alkalinity									
	Aqueous	SM2320	Polyethylene bottle	100mL	4°C	24 hours			
Ammonia									
Chlorid	Aqueous	SM4500NH3B	Polyethylene bottle	100mL	4°C H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days			
Cilioria	Aqueous	SM4500Cl B	Polyethylene bottle	100mL	4°C	28 days			

Table 7-3 (cont'd)

# Recommended Containers, Preservation Techniques and Holding Times for Other Analyses

Analyte COD	<u>es</u>	Method	Containers	Required Volume	Preservation	Holding Times
	Aqueous	SM5220C, D	Amber glass bottle	50mL	4°C H2SO4, pH<2	28 days
Color						
	Aqueous	SM2120B	Polyethylene bottle	50mL	4°C	Immediate
Nitrates	S					-
	Aqueous	SM4500NO3 E	Polyethylene bottle	50mL	4°C H₂SO₄, pH<2	48 hours 7 days
Nitrite						
	Aqueous	SM4500NO2 b	Polyethylene bottle	50mL	4C	48 hours
Orthopl	hosphate		•			
	Aqueous	SM4500-P, E	Polyethylene bottle	50mL	4°C	48 hours
Total pl	hosphate			,		
	Aqueous	SM4500-P B, E	Polyethylene bottle	50mL 50mL	4°C HCl, pH<2	24 hours 28 days
Phenols	:					
	Aqueous	SM5530B SM5530C	Polyethylene bottle	250mL	4°C H₂SO₄, pH<2	28 days
Sulfates	i					
	Aqueous	SM4500SO4 E	Polyethylene bottle	50mL	4°C	28 days
Sulfide Total						
	Aqueous	SM4500-S D	Polyethylene bottle	50mL	4°C	28 days
Reactivi	itv					
	Solid	Chapter 7 SW846	Amber glass jar	10gram	4°C	28 days
	Aqueous		Polyethylene bottle	250mL	4°C	28 days
Total Or	ganic Carbon (To	•				
	Solid	9060	Amber glass jar	20g	4°C	14 days
	Aqueous	415.1	40mL VOA vials	40mL	4°C HCl, pH<2	28 days

### Table 7-3 (cont'd)

# Recommended Containers, Preservation Techniques and Holding Times For Other Analyses

Analytes TKN	Method	Containers	Required* Volume	Preservation	Holding <u>Times</u>
Aqueous	SM4500Norg C	Polyethylene bottle or Amber glass bottle	50mL	4°C H₂SO₄, pH<2	28 days
Total Solids (TS)					
Aqueous	SM2540B	Polyethylene bottle	200mL	4°C	7 days
Total Dissolved Solids	(TDS)				
Aqueous	SM2540C	Polyethylene bottle	200mL	4°C	7 days
Total Suspended Solids	(TSS)				
Aqueous	SM2540D	Polyethylene bottle	200mL	4°C	7 days
Settleable Solids					
Aqueous	SM2540F	Polyethylene bottle	200mL	4°C	48 hours

<sup>\*</sup> These represent minimum required volume. Additional sample volumes should be collected to minimize headspace loss for volatile analysis. Additional sample aliquot are also required to perform QA/QC functions (e.g. spikes, duplicates), % moisture for solid samples and sample re-analysis (if needed).

<sup>&</sup>lt;sup>a</sup> For Massachusetts analyses, the volatile soil samples are to be preserved in methanol in the field.

#### 8.0 SAMPLE CUSTODY

### 8.1 Chain of Custody:

Samples are physical evidence collected from a facility or the environment. In hazardous waste investigations, sample data may be used as evidence in (EPA) enforcement proceedings. In support of potential litigation, laboratory chain-of-custody procedures have been established to ensure sample traceability from time of receipt through the disposal of the sample.

A sample is considered to be in the custody under the following conditions:

- It is in an authorized person's actual possession, or
- It is in an authorized person's view, after being in that person's physical possession, or
- It was in an authorized person's possession and then was locked or sealed to prevent tampering, or
- It is in a secure area.

Chain-of-custody originates as samples are collected. Chain-of-custody documentation accompanies the samples as they are moved from the field to the laboratory with shipping information and appropriate signatures indicating custody changes along the way.

Laboratory chain-of-custody is initiated as samples are received and signed for by the Sample Custodian or his/her designate at MITKEM. Documentation of sample location continues as samples are signed in and out of the central storage facility for analysis in the several MITKEM departments using the Sample Tracking Forms (Fig 8.4-1). After analysis, any remaining sample is held in the central storage area to await disposal.

### 8.2 Laboratory Security:

Samples at MITKEM are kept within the secure areas during all stages of residence, including the periods of time spent in preparation for analysis, while undergoing analysis and while in storage.

The entire laboratory is designated as a secure area. The doors to these areas are under continuous surveillance or are kept locked after regular business hours and may only be accessed by key. Only authorized personnel are allowed to enter the secure areas. Visitors to the laboratory must be accompanied by MITKEM staff members.

8.3 Duties and Responsibilities of Sample Custodian:

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Duties and responsibilities of the Sample Custodian include but are not limited to:

- 8.3.1 Receiving samples.
- 8.3.2 Inspecting and documenting sample shipping containers for presence/absence and condition of:
  - 8.3.2.1 Custody seals, locks, "evidence tape", etc.;
  - 8.3.2.2 Container breakage and/or container integrity.
- 8.3.3 Recording condition of both shipping containers and sample containers (cooler temperature, bottles, jars, cans, etc.).
- 8.3.4 Signing Documents shipped with samples (i.e. air bills, chain-of-custody record(s), Sample Management Office (SMO) Traffic Reports, etc.)
- 8.3.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e. sample tags, chain-of-custody records, traffic reports, air bills, etc.). If there is non-agreement, recording the problems, contacting the client for direction, and notifying appropriate laboratory personnel. (Client's corrective action directions shall be documented in the case file.)
- 8.3.6 Initiating the paper work for sample analyses on laboratory documents (including establishing sample project files) as required for analysis or according to laboratory standard operating procedures.
- 8.3.7 Label samples with laboratory sample identification numbers, and cross-referencing laboratory numbers to client numbers and sample tag numbers.
- 8.3.8 Placing samples and spent samples into appropriate storage and/or secure areas.
- 8.3.9 Controlling access to sample in storage and assuring laboratory standard operating procedures are followed when samples are removed from and returned to storage.
- 8.3.10 Where applicable, making sure that sample tags are removed from the sample containers and included in the project file.

- 8.3.11 Where applicable, accounting for missing tags in a memo to the file or documenting that the sample tags are actually labels attached to sample containers or were disposed of, due to suspected contamination.
- 8.3.12 Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination.
- 8.3.13 Sending shipping containers, prepared sample bottles and sample instructions to clients who request them.
- 8.3.14 Recording temperatures of freezers and refrigerators in the laboratories.

### 8.4 Sample Receipt:

Sample shipments are received at MITKEM by the Sample Custodian or his/her designated representative. Unless the shipment is a continuation of a previous project, a new project file is started for the sample. The information is logged into the Sample Receipt Logbook (Figure 8.4-1).

The cooler is inspected for the following (if applicable) and documented on the Sample Login Form (Figure 8.4-2) for USEPA CLP samples and on the Sample Condition Form (Figure 8.4-3) for the other samples:

- Custody seal (conditions and custody number)
- Air bill (courier and air bill #)

The cooler is then opened and the following checked (in order). Make sure the hood is turned of when the cooler is opened.

- Chain of custody record (or traffic report). These are usually taped to the inside of the cover.
- Cooler temperature (use temperature blank if available) using the temperature gun.
- Coolant condition (e.g. ice intact, melted)
- Radioactivity using the Geiger counter.

The Sample Custodian will perform the following:

- Remove the sample containers and arrange them in the same order as documented in the chain of custody report.
- Inspect condition of the sample containers.
- Assign laboratory sample ID and cross reference the laboratory ID to the client ID.
- Remove tags and place in the project file.
- Check preservative and document in the Sample Condition Form (Figure 8.4-3) if needed.
- Peer review to ensure proper cross referencing and labeling of sample containers.

Any discrepancies or problems are noted in the Sample Condition Notification Form (Figure 8.4-4).

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Depending on the project, the sample custodian may directly inform the client of the discrepancies or convey the information to the project manager who will in turn inform the client.

Following the resolution of any problems or discrepancies, the Sample Custodian signs the Sample Receipt Form and originates a file for the set of samples, including in it the Sample Receipt Form, chain of custody records, and shipping information.

When the Sample Custodian is not available to receive samples, the sample container is signed for by another MITKEM staff member, the time, date and name of the person receiving the container are recorded on the custody records. In addition, if present, custody seal number is recorded and the cooler temperature is measured and recorded on the Sample Condition Form. The samples are then stored in the centralized walk-in refrigerator in the sample receipt area. The sample receipt area is located in the secure area of the laboratory. The samples are officially received and documented by the Sample Custodian or designee before the next business day.

At times, samples will be sent to another lab for analysis not performed at MITKEM. These subcontracted analyses will be performed by laboratories that are certified to perform the analyses. These samples are placed in bubble bags to prevent breakage and stored in a cooler with ice. The samples are either hand delivered to a local sub-contract lab or air courier with MITKEM chain-of-custody (Figure 8.4-5).

### 8.5 Sample Log-in Identification:

### 8.5.1 Sample Identification;

To maintain sample identity, each sample received at MITKEM is assigned a unique sample identification (Sample ID) number. Samples are logged into MITKEM via the ChemWare Horizon Laboratory Information Management System (LIMS).

After inspecting the samples, the Sample Custodian assigns each sample a MITKEM Sample ID Number. These Numbers are assigned sequentially in chronological order. MITKEM Sample Identification Numbers appear in the following format: Wxxxxyyy

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where: W – represents the current year with 6 for 1999

xxxx – represents a four digit project number which is assigned sequentially

yyy – represents the sample number within the group or case.

For example, the fifth sample of the 20<sup>th</sup> case of 1999 would have the number: 60020005

The MITKEM Sample ID Numbers are recorded on the Sample Login Form (Figure 8.4-2) for USEPA CLP samples and on the Sample Condition Form (Figure 8.4-3) for the other samples. Information on these forms cross - reference the Sample ID Numbers with SDG numbers, sample tag numbers and/or other client identifiers. Each sample is clearly labeled with its MITKEM Sample ID Number by the Sample Custodian. The same sample ID Number appears on the LIMS status report, on each sample preparation container and extract vial associated with the sample.

### 8.5.1.1 Sample Extract Identification:

As described in Section 8.5.1, a sample extract is identified with the same unique sample identification number as the sample from which it derives. In addition, it bears one of the following prefixes:

### For Organic Analyses:

S for Semivolatile Organics
F for TPH
EPH for Extractable Petroleum Hydrocarbons
O&G for Oil and Grease
H for Herbicides
P for Pesticides
B for PCBs
P is also used for CLP analysis when Pesticide and PCB are analyzed as a single analysis.

#### For Inorganic Analyses:

I for ICAP analysis
Z for Zeeman Graphite Furnace analysis
C for Cyanide analysis
N for Ammonia analysis
S for Sulfite analysis

### 8.5.2 Sample Login:

Sample login system at MITKEM consists of computerized entry using ChemWare Horizon LIMS (Figure 8.5-1). The information recorded onto the Project Information Form includes:

• Project number

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- Client name
- QC requirements
- Date of receipt
- Date sample collected
- Due date
- Initials of the Sample Custodian and Project Manager
- Comments
- MITKEM Sample Identification numbers
- Client Sample Identification numbers
- Sample matrix
- Analyses required
- Cost of analyses
- Reporting requirements

### 8.5.3 Sample Information:

After sample information is properly recorded (Sample Receipt Logbook, Sample Receipt Forms and Project Information Forms) and samples have properly been assigned Sample ID numbers and labeled, the Sample Custodian notifies the Project Manager. The Project Manager reviews all the information associated with the samples. He/she verifies (by dating and initialing) the correctness of the information on the Project Information Form.

A project file is initiated by the Sample Custodian. This file contains the original Sample Project Form, air bills, SMO traffic reports and all correspondence with the Client or SMO of others.

Copies of the Project Information Forms are distributed to the various departments.

### 8.6 Sample Storage and Disposal:

Samples at MITKEM are stored in a central storage facility. After sample receipt and login procedures are completed, the Sample Custodian places the samples in the centralized walk-in refrigerator. Volatile Organic sample aliquots are released to the volatile organic lab with documentation (Figure 8.6-1).

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The sample storage area are for samples only, no standards or reagents are to be stored there. Access to the centralized sample storage area is locked at all times.

All sample/extract refrigerators are maintained at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Standards are kept in freezers maintained at -10 to -20°C. Temperatures are recorded every working day in the Temperature Log (Figure 8.6-2). Corrective action for refrigerator and/or freezer temperatures; outside of the acceptable ranges are posted on the refrigerators and/or freezers.

When analysis is complete, any remaining sample is retained in the central storage area until it may be removed for disposal (see SOP G24 for Sample Disposal). Broken and damaged samples are promptly disposed of in a safe manner. Unless notified by the client, excess, unused sample aliquots are stored for at least 30 days after the submission of compliant data. The samples are then disposed after such period. USEPA and NYS ASP extracts are stored for at least one year. The rest of the sample extracts are stored for at least 30 days after data submission. After such time, the extracts are disposed of. All disposals are documented in a manner compliant with federal and state regulations.

#### 8.6.1 Extract Transfer

The extracts generated during the preparation for the organic analyses are transferred from the Organic Prep Lab to the Analysis Labs. The extracts, for Semivolatiles, TPH, Pesticides and PCBs, are checked in the Analysis Lab by entries in the appropriate Extract Transfer Logbook (Figures 8.6-3 and 8.6-4)

### 8.6.2 Extract Storage:

Semivolatile, Pesticide/PCB, and TPH extracts, which are contained in crimp top vials or screw cap vials with Teflon lined septa, are stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ . Semivolatile extracts are stored in R7. Pesticide/PCB and TPH extracts are stored in R5. They are catalogued numerically by project number that approximates chronological order, according to date of receipt. USEPA CLP extracts are stored separately from sample extracts of other clients.

Excess Pesticide extracts, not analyzed, are stored in screw cap vials with Teflon lined septa in the Organic Prep Lab. In most instances, they consist of the remaining 8.5mL portions of aqueous and soil sample extracts and are chronologically ordered.

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### 8.7 Sample Tracking:

When a sample is removed from storage, the analyst who has custody signs the Sample Tracking Log. This information indicates the location of the sample at any point in time.

Chain-of-custody of a sample ensures that the sample is traceable from when it was taken in the field through laboratory receipt, preparation, analysis and finally disposal. The primary chain-of-custody documents are used to locate a sample at any point in time.

- 1. The chain-of-custody form from the field describing the origin and transportation of a sample;
- 2. The laboratory Sample Receipt Log (Sample Tracking Log) and supporting login records, documenting acceptance of a sample by the Mitkem laboratory; and
- 3. The MITKEM Sample Tracking Log, documenting which analyst has custody of the sample after removal from storage.

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## Figure 8.4-1 Sample Receipt Logbook (Sample Tracking Logbook)

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## MITKEM SAMPLE RECEIPT LOG BOOK

	Date		Storage	#'s/Analyst/Date						
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### Figure 8.4-3 Sample Condition Form

## MITKEM CORPORATION Sample Condition Form

Page\_of\_

Received By:	Reviewed By:		Date <sup>-</sup>		-			М	ITKEM	Project	
Client Project:			Client								
			Sample ID					(pH)		Comments/Remark	ks/
Condition:		Lab	Client	- 1	1NO3	H2SO-	H	⊠ Na	ОН	Corrective Action	1
				- -	_		_				
1) Custody Seal(s)	Present/Absent										
•	Coolers/Bottles			_ _			_				
	Intact/Broken			_ _							
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2) Custody Seal Number(s)	<del></del>										
				_ _				<u>                                     </u>			
3) Chain-of-Custody	Present/Absent										
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4) Cooler Temperature				$\perp$							
Coolant Condition											
			<del></del>							•	
5) Airbill(s)	Present/Absent										
Airbill Number(s)											
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) Sample Bottles	Intact				-						
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	Leaking										
Date Received											
Time Received											
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Project Due Date					T	1	$\exists$				
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Figure 8.4-2 USEPA CLP Sample Login Form

### Figure 8.4-2 USEPA CLP Sample Login Form

	Lab Hemx MITICEM	CORP				Pageof
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þ	Leceived By (Signature)					
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R	courts: ,			Con	reponding	
			EPA Sample 1	Sample Tag 1	Assigned Lab 1	Remets Coadition of S Shipment, e
1.	Custody Scal(s)	Present/Absent* Intact/Broken			·	
2.	Curody Seal Nov.		-			
3.	Chain-of Custody Records	Present'Absent*				
۷.	Treffic Reports or Packing Lists	Present'Absent*				
5.	Aidill	Airbill/SticLer Present Absent				
s	Airbill No		-			
1.	Sample Tags	Present/Absent*				
	Sample Tag Numbers	Liacd/Not Listed on Chain- of-Custedy				
i.	Sample Condition	Intest/Broken*/Leaking				
1.	Docs information on custody records.  traffic reports, and sample tags agree?	Yes'Na*				
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	Sample Transfer					
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Figure 8.4-3 Sample Condition Form

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### Figure 8.4-3 Sample Condition Form

## MITKEM CORPORATION Sample Condition Form

Page\_of\_

Received By:	Reviewed By:		Date:			MITK	MITKEM Project				
Client Project:			Client								
			Sample ID		Preservation (p			Comments/Remarks/			
Condition:		Lab	Client	HNC	HUSO	ч на	NaOH	<del></del>			
ι								•			
1) Custody Seal(s)	Present/Absent										
•	Coolers/Bottles										
	Intact/Broken										
				-	-						
2) Custody Seal Number(s)					1						
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Coolant Condition				<b> </b>							
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Airbill Number(s)							_  .				
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## Figure 8.4-4 Sample Condition Notification Form

### Figure 8.4-4 Sample Condition Notification Form

Sample Condition Notification		Page	of
cample condition Notification			
Mitkem Project#:	Date of Receipt:_		
Client:	Received By:		
Client project #/name:			
Unusual Occurance Description:			
		····	
•			
Client Contacted:			
Contacted via: Phone/Fax			
Date:Time:			
Contacted By:		w	•
Name of person contacted:		_	
Client Response:			
Responded via: Phone/Fax			
Date:			•
Name of person responding:			
Responding to:			
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litkem Action Taken:			

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Figure 8.4-5
MITKEM Chain-of-custody Form



175 Metro Center Boulevard Warwick, Rhode Island 02886-1755 (401) 732-3400 • Fax (401) 732-3499 email. mitkem@worldnet att net

## CHAIN-OF-CUSTODY RECORD

Page \_\_\_\_ of \_\_\_\_

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Figure 8.4-5
MITKEM Chain-of-custody Form

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Figure 8.5-1
Project Information Form

## MITKEM CORPORATION

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Revision #1

Logged in By:

Reviewed By:

Date Opened: Date Closed:

Project Status

Lab Workorder #: 60322

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INVOICE GOES TO:

Lab ID 60322001	Client ID	<u>Matrix</u> W	Analysis Code 418.1WTRPH 8260W	Collected 02/28/99 07:45	<u>Received</u> 03/09/99	<u>Due</u> 03/16/99	Notes
60322002		W	8260W	03/01/99 16:30	03/09/99	03/16/99	
60322003		W	418.1WTRPH 8260W			03/16/99	
60322004		W	418.1WTRPH 8260W 8270W RCRA8W	03/02/99 06:00	03/09/99	03/16/99	RCRA8 metals are dissolved
60322005			418.1WTRPH 8260W 8270W RCRA8W	03/02/99 15:00	03/09/99	03/16/99	RCRA8 metals are dissolved

REPORT GOES TO:

Figure 8.5-1 Project Information Form

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Figure 8.6-1 Volatile Organic Tracking Logbook

## 

## MITKEM CORPORATION: VOLATILE ORGANICS TRACKING LOGBOOK

LAB ID	CLIENTID	DATE/TIME IN	ANALYS	DATE/TIME OUT	DATE/TIME IN	ANALYS	DATE/TIME OUT	DATE/TIME IN	ANAI VO	COMMENTS
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Figure 8.6-1 Volatile Organic Tracking Logbook

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Figure 8.6-2 Temperature Log

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MITKEM CORPORATION TEMPERATURE LOGBOOK

Temperature Requirements: Refrigerators between 2 and 6 C Freezers between -10 and -20 C

Month/Year:

	Time	R1	R2	R3	R4	R5	R6	R7	R8	R9	R10	R11	R12	R13	F3	F4	F5	F7	F9	F40	lla lata i
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Figure 8.6-2
Temperature Log

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## Figure 8.6-3 Extracts Transfer Logbook – Semivolatile Analysis

Figure 8.6-3
Extracts Transfer Logbook – Semivolatile Analysis

Date Transferred		Transferred	Received	Storage	
from Prep Lab	Lab ID	by	by	Location	Comments
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## Figure 8.6-4 Extracts Transfer Logbook – Pesticide/PCB Analysis

## Figure 8.6-4 Extracts Transfer Logbook – Pesticide/PCB Analysis

MITKEM CORPORATION EXTRACTS TRANSFER LOGBOOK: PESTICIDES/PCB ANALYSIS								
Date Transferred rom Prep Lab	Lab ID	Transferred by	Received by	Storage Location	Comments			
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### 9.0 CALIBRATION PROCEDURES AND FREQUENCIES

#### 9.1 Instruments:

Specific calibration and check procedures are given in the analytical methods referenced in Section 10. The frequencies of calibration and the concentrations of calibration standards are determined by the cited methods and special contractual requirements. Standard calibration curves of signal response versus concentration are generated on each analytical instrument used for a project, prior to analysis of samples. A calibration curve of the appropriate linear range is established for each parameter that is included in the analytical procedure employed and is verified on a regular basis with check standards as specified in the appropriate CLP Protocols. For non-CLP work, MITKEM adheres to the calibration criteria specified by SW-846 and or Standard Methods for both organic and inorganic analyses. Where requested, other method specific calibration criteria are used.

The following are examples of calibration procedures for various instrumental systems. Please refer to the Standard Operating Procedures for the specific calibration requirements.

GC – An initial calibration is performed using five different concentration levels for each parameter of interest for SW-846 analyses. The initial calibration is done on each column and each instrument, and is repeated each time a new column is installed or whenever a major change is made to the chromatographic system.

An initial calibration verification (ICV), near mid level concentration for all analytes, is performed immediately after the calibration. If the ICV does not meet method specific criteria, a new calibration curve is generated and an ICV is run. If repeated ICV failures are encountered, the system is checked out to find the cause of these failures and the problem is corrected.

A continuing calibration verification (CCV), near a mid - level concentration for all analytes, is run at ten (10) sample intervals. If CCV values are determined outside the upper limit of the method specified range and if no analytes were detected in the samples, the run will be accepted as valid and 'No Detects' reported for the sample. If an analyte is detected and the CCV is out at the high end, the problem will be identified and corrected and the affected samples will be re-analyzed with a compliant CCV.

If a CCV value is out of the method specified limits at the lower limit, the cause of the problem will be identified and corrected, and all samples affected by the out of control CCV will be rerun with a compliant CCV.

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For CLP-type analyses, the continuing calibration takes place at the beginning of the analytical sequence and once every twelve (12) hours throughout the analytical sequence. The percent difference in calibration factors for each standard must not exceed the criteria specified by the method.

If a CCV fails to meet criteria limits, a new calibration curve will be generated and all samples affected will be re-analyzed.

GC/MS – For CLP methods, a minimum of five level calibration (four level for selected semivolatile compounds) is carried out for each analyte per system before analysis of samples take place.

Continuing calibrations, near midpoint levels, are analyzed every twelve hours of instrument analysis time for CLP analyses.

Re-calibration takes place whenever a major change occurs in the system, such as a column change in the GC or a source cleaning of the mass spectrometer or when the continuing calibration fails to meet method specific requirements.

Tunes are performed once every twelve (12) hours. The GC/MS system is tuned to USEPA specifications for bromofluorobenzene (BFB) or decafluorotriphenylphosphine (DFTPP) for volatile and semivolatile analyses, respectively. Venfication of tuning criteria occurs every twelve hours of instrument run time for all CLP-type and SW846 analyses.

More detailed instrument and method-specific calibration procedures and criteria are described in the individual analysis SOPs.

ICAP – Instrument calibration, for each wavelength used, occurs at the start of each analysis. The calibration curve is constructed per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed after calibration.

During the analysis, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed every ten (10) samples. If either the CCV or CCB fails to meet method specific criteria for an analyte, the analyte is reanalyzed after calibration.

The CCV is obtained from a source independent from that of the standards. The CCV concentration for the different analytes are at method specified levels.

GFAA (Zeeman Graphite Furnance) – At the start of each analysis, the instrument is calibrated per method specification.

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An initial calibration verification (ICV) and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed after re-calibration.

During the analyses, a continuing calibration verification (CCV) and continuing calibration blank (CCB) is analyzed every ten (10) samples. If either the CCV or CCB fails to meet method specific criteria for an analyte, the analyte is reanalyzed after calibration.

The CV is obtained from a source independent from that of the standards. The CV concentration for the different analytes are at method specified levels.

### Other instrumentation:

pH meter is calibrated at three pH levels (4.0, 7.0, and 10.0) before analyses of samples.

Lachat 8000 automated flow-through spectrophotometer is calibrated per method specification before the analyses of samples.

An initial calibration verification and initial calibration blank are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed after re-calibration.

During the analyses, a continuing calibration verification and continuing calibration blank is analyzed every ten (10) samples. If either the CCV or CCB fails to meet specified criteria for an analyte, the analyte is re-analyzed after recalibration.

The CCV is obtained from a source independent from that of the standards. The CV concentration for the different analytes are at method specified levels.

Spectronic 20 manual spectrophotometer is calibrated per method specification.

An initial calibration verification and initial calibration blank (ICB) are analyzed before analysis of samples. If the ICV and ICB do not meet method specific criteria for an analyte, the analyte is re-analyzed after re—calibration.

During the analyses, a continuing calibration verification and continuing calibration blank are analyzed every ten (10) samples. If either the CCV or CCB fails to meet method specified criteria for an analyte is re-analyzed after recalibration.

The CV is obtained from a source independent from that of the standards. The CV concentration for the different analytes is not method specific levels.

Balances are calibrated once a year by an outside service.

A calibration check is performed with NIST verified weights quarterly.

Class "S" weights are NIST certified by an outside certified service every 2 years.

A verification check is performed with Class 3 weights each day.

Thermometers are calibrated once a year against a NIST verified thermometer.

The NIST thermometers are certified by an outside certified service annually.

### 9.2 Standards and Reagents:

Standard reference materials used for routine calibration, calibration checks, and accuracy are obtained from commercial manufacturers. These reference materials are traceable to the source and readily compared to EPA references. Certain projects, especially those involving pesticide registration, may necessitate the use of reference standards supplied by the client. New standards are routinely validated against known standards that are traceable to EPA or NBS reference materials.

Standards are dated upon arrival. Any material exceeding its shelf life as described by the methods in Section 10.0 is discarded and replaced. Standards are periodically analyzed for concentration changes and inspected for signs of deterioration such as color change and precipitate formation. Standards Receiving and Preparation Logbooks, which contain all pertinent information regarding the source and preparation of each analytical standard, are maintained by each of the MITKEM laboratory departments (Figures 9.2-1 to 9.2-4).

Solvents and acids are examined for purity prior to use to ensure there is no external source of contamination.

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Figure 9.2-1
Metals Primary Standard Receipt Logbook – Instrument Laboratory

Date Received	Primary STD ID	Vendor	Catalog #	Lot#	STD RECEIPT	Received	Exp.	Disposal or	Milkem
					Allalytes	Ву	Date	Consumption Date	ID
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Figure 9.2-2 Semivolatile Primary Standard Logbook – Preparation Laboratory

	1			MOTIVET. IT	CHMART 31.	ANDARD LOGB	OUK		
Date Rec	Primary STD ID	Vendor	Catalog #	Lot#	Conc.	Analyte(s)	Rec. By	Date Opened	Expir Date
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Figure 9.2-3
Pesticide/PCB Working Standard Logbook – Preparation Laboratory

		ON PESTICIDES WORKIN			1/11/	NOTION	LAB		
PREP DATE		PRIMARY/INTERMEDIATE STD ID	INITIAL CONC.	AMT. ADDED	FINAL VOL	FINAL CONC.	SOLVENT		PREPO
					101	CONC.	. USED	WORKING STD ID	BY
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Figure 9.2-4
Reagent Preparation Logbook – Preparation Laboratory

## Mitkem Corporation Organic Preparation Laboratory Reagent Preparation Logbook

Reagent	Manufacturer	Lot#	Conc.	Dilution	Final Conc.	Exp. Date	Analyst
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Figure 9.2-4
Reagent Preparation Logbook – Preparation Laboratory

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## 10.0 ANALYTICAL PROCEDURES

MITKEM uses the methods specified in Tables 10-1 through 10-6 unless otherwise specified by the client.

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## Table 10-1 Potable Water Analytical Methods

Parameter	Method Description	Method Reference
Metals  Aluminum, Barium, Chromium, Cobalt, Copper, Iron, Manganese, Molybdenum, Nickel, Silver, Strontium, Silver, Titanium, Vanadium, Zinc	ICAP Analysis*	200.7
Mercury	Cold Vapor Analysis	245.1
Residual Chlorine	Spectrophotometric	SM4500-C1 G
Trihalomethanes	Purge&Trap GC/MS Analysis	524.2
Volatile Organic Compounds	Purge&Trap GC/MS Analysis	524.2
1.2-Dibromo-3-chloropropane 1,2-Dibromomethane	Micro extraction GC Analysis	504.1

<sup>\*</sup> Please note; Antimony, Arsenic, Beryllium, Cadmium, Lead, Selenium, and Thallium analyses in potable water are subcontracted to a Drinking Water certified laboratory. Mitkem is not certified for Graphite Furnace Atomic Absorption (GFAA) analysis for these analytes.

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## Table 10-2 Non-potable Water Priority Pollutant Analytical Methods

Parameter Metals  Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Iron, Lead, Manganese, Molybdenum, Nickel, Selenium, Silver, Strontium, Silver, Thallium, Titanium, Vanadium, Zinc	Method Description ICP	Method Reference 200.7
Mercury	Cold Vapor	245.1
Alkalınity	Titration	SM2320
Chloride	Titration	SM4500-C1 B
рН	Electrode	SM4500 H+ B
Sulfate	Spectrophotometric	SM4500-SO4 E
Ammonia	Distillation/Nesslerization	SM4500-NH3 B
Nitrate	Cadmium reduction	SM4500-NO3 E
Nitrite		SM4500-NO2 B
Orthophosphate	Ascorbic, Manual	SM4500-P E
Total phosphate	Persulfate, Manual	SM4500-P B3 & E
Chemical Oxygen Demand	Spetrophotometric	SM5220-C, D
Total Organic Carbon	Combustion	415.1
Phenolics	Distillation, Color, Automated	SM5530 B
Total Dissolved Solids	Gravimetric	SM2540 C
Total Solids	Gravimetric	SM2540 B
Total Suspended Solids	Gravimetric	SM2540 D

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Table 10-2 Non-potable Water Priority Pollutant Analytical Methods (con't)

Parameter	Method description	Method Reference
Total Settleable Solids	Imhoff cones	SM2540 F
Volatile Organics Halocarbons Aromatics	Purge & Trap, GC/MS Purge & Trap, GC/MS	624 624
Semivolatile Organics	Extraction, GC/MS	625
Organochlorine Pesticides/ PCBs	Extraction, GC	608
Oil & Grease	Extraction, Gravimetric	1664

## Table 10-3 SW-846 Inorganic Analytical Methods

Parameter Metals	Method Description	Method Reference
Aqueous	Acid digestion ICAP analysis	Method 3005A/3010A Method 6010B
Solid	Acid digestion ICAP analysis	Method 3050A Method 6010B
Mercury		
Aqueous	Permanganate digestion Cold Vapor analysis	Method 7470
Solid	Permanganate digestion Cold Vapor analysis	Method 7471
Hexavalent Chromium		
Aqueous	Diphenyl Carbazide Colorimetric	Method 7196
Solid	Acid Digestion Colormetric	Method 7196
Cyanide		
Aqueous	Midi-distillation Automated	Method 9012A
Solid	Midi-distillation Automated	Method 9012A
pH		
Solid	Electrode	Method 9045
Ignitability (Flashpoint)		
Aqueous	Pensky-Martens closed cup	Method 1010
Solid	Pensky-Martens closed cup	Method 1010
Reactive Cyanide Solid	Midi-distillation Automated	7.3.3.2
Reactive Sulfide		
Solid	Colorimetric	7.3.3.4

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# Table 10-3 SW-846 Inorganic Analytical Methods (con't)

Parameter	Method Description	Method Reference		
Toxicity Characteristic Le	aching Procedure (TCLP)			
Aqueous	Method 1311	Method 1311		
Solid	Method 1311	Method 1311		
Synthetic Precipitation Leaching Procedure (SPLP)				
Aqueous	Method 1312	Method 1312		
Solid	Method 1312	Method 1312		

## Table 10-4 SW-846 Organic Analytical Methods

Parameter Volatile Organic Compounds	Method Description	Method Reference			
Aqueous	Method 5030	Method 8260B			
Solid	Method 5030 Method 5035	Method 8260B			
Volatile Organic Compounds (Aron Aqueous	natic + Methyl t-butyl ether (MTBE)) Method 5030	Method 8021B			
Solid	Method 5030	Method 8021B			
Semivolatile Organic Compounds Aqueous	Method 3510C Method 3520C	Method 8270C			
Solid	Method 3540C Method 3550B	Method 8270C			
Organochlorine Pesticides					
Aqueous	Method 3510C Method 3520C	Method 8081A			
Solid	Method 3540C Method 3550B	Method 8081A			
Polychlorinated Biphenyls					
Aqueous	Method 3510C Method 3520C	Method 8082			
Solid	Method 3540C Method 3550B	Method 8082			
Total Petroleum Hydrocarbons					
Aqueous	Method 3510C Method 3520C	Method 8015M			
Solid	Method 3540C Method 3550B	Method 8015M			

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## Table 10-4 SW-846 Organic Analytical Methods (con't)

Param Herbio		Method Description	Method Reference
Heron	Aqueous	Method 8151A	Method 8151A
	Solid	Method 8151A	Method 8151A
Toxic	ity Characteristic Leaching Pro Aqueous	ocedure (TCLP) Method 1311	Method 1311
	Solid	Method 1311	Method 1311
Synthe	etic Precipitation Leaching Pro Aqueous	ocedure (SPLP) Method 1312	Method 1312
	Solid	Method 1312	Method 1312
Gel Pe	ermeation Chromatography (G Aqueous	PC) Method 3640A	Method 3640A
	Solid	Method 3640A	Method 3640A
Floris	il Cleanup Aqueous	Method 3620B	Method 3620C
	Solid	Method 3620B	Method 3620B
Silica	Gel Cleanup Aqueous	Method 3630C	Method 3630C
	Solid	Method 3630C	Method 3630C
Sulfur	Cleanup Aqueous	Method 3660B	Method 3660B
	Solid	Method 3660B	Method 3660B
Sulfur	ric Acid Cleanup Aqueous	Method 3665A	Method 3665A
	Solid	Method 3665A	Method 3665A

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## Table 10-5 CLP-Type Analytical Methods

Parameter USEPA CLP Organics

Method Reference

OLM04.2

USEPA CLP Inorganics

ILM04.1

USEPA Low Level Organics

OLC02.1

NYS-ASP CLP Organics

ASP '95 SOW

NYS-ASP CLP Organics

ASP '95 SOW

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## Table 10-6 Other Analytical Methods

Parameter	Method Description	Method Reference			
Volatile Petroleum Hydrocarl	oons				
Aqueous	MADEP VPH 98-1	MADEP VPH 98-1			
Solid	MADEP VPH 98-1	MADEP VPH 98-1			
Extractable Petroleum Hydrocarbons					
Aqueous	MADEP EPH 98-1	MADEP EPH 98-1			
Solid	MADEP EPH 98-1	MADEP EPH 98-1			
New York State Total Petroleum Hydrocarbon					
Solid	310.13	310.13			

#### 10.1 Analytical References

- 1. Analytical Services Protocol, Volume 1-8, New York State Department of Environmental Conservation, September, 1989.
- 2. Annual Book of ASTM Standards. Part 31-Water. American Society for Testing and Materials, Philadelphia, PA, 1981.
- 3. Chemical Characteristics of Marine Samples, API Publications No. 4307, API, Washington, D. C.
- 4. Federal Register. Vol. 55, No. 61, March 29, 1990
- 5. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 3/83 Revision.
- 6. The EPA 600 Series. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, Appendix A, 40 CFR Part 136, Federal Register, Vol. 49, No. 209, 1984.
- 7. Methods of Soil Analysis. Part 2, Chemical and Microbiological Properties, Second Edition, American Society of Agronomy, Inc., Soil Science Society of America, Inc., Madison, WI, 1982.
- 8. Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, APHA, Washington, D. C., 1992.
- 9. Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, SW-846, 3<sup>rd</sup> Edition Update III. Office of Solid Waste and Emergency Response, USEPA, Washington, D. C., 1996.
- 10. USEPA Contract Laboratory Program. Statement of Work for Organic Analysis, USEPA, OLM04.2.
- 11. USEPA Contract Laboratory Program. Statement of Work for Inorganic Anlaysis, USEPA, ILM04.1.

#### 11.0 DATA REDUCTION, VALIDATION AND REPORTING

#### 11.1 Data Reduction:

Instrument print-outs, computer terminal displays, chromatograms, strip chart recordings and physical measurements provide raw data that are reduced to concentrations of analytes through the application of the appropriate calculations.

Equations are generally given within the analytical methods referenced in Section 10. Data reduction may be performed automatically by computerized data systems on the instrument, manually by the analyst, or by PCs using spreadsheet and/or data base software. This software includes Thru-Put's 'TARGET' for the analyses of organic analytes and Ward Scientific 'EDR' for metals, cyanide and mercury analysis.

#### 11.2 Data Validation:

Data validation is an essential element of the QA evaluation system. Validation is the process of data review and subsequent acceptance or rejection based on established criteria.

The following analytical criteria are employed by MITKEM in the technical evaluation of data:

- Accuracy requirements
- Precision requirements
- Detection limit requirements
- Document requirements

As in the case of EPA/CLP procedures, data acceptance limits may be defined within the method. As one means of tracking data acceptability, quality control charts will be plotted for specific parameters determined in similar, homogeneous matrices. Control limits for methods are statistically determined as analytical results are accumulated.

Upon completion of the evaluation, the evaluator dates and initials the data review checklist as described in Section 11.5 below.

#### 11.3 Data Verification:

The verification process requires the following checks to be made on data packages before they are submitted to the client:

- A completeness inspection is required which ensures that all required data are
  included in the data packages submitted to the client and that the appropriate
  signatures are present on the data packages.
- A contract compliance screening to ensure that contractual requirements have been satisfied.
- A consistency check to ensure that nominally identical or similar data appearing in different places within a data package are consistent with respect to value and units.
- A correctness check to ensure that reported data have been calculated correctly or transcribed correctly.

#### 11.4 Data Interpretation and Reporting:

Interpretation of raw data and calculation of results are performed by a scientist experienced in the analytical methodology. Upon completion of data reduction, the scientist signs for the reported results on the data report narrative.

The laboratory supervisor, who is responsible for the data generated in that department, performs an independent review of data and completed report forms. Members of the QA staff also check the results on selected sets of data (usually 10%).

#### 11.4.1 Report Formats:

Two types of data reports are generated at Mitkem: commercial data reports and CLP data reports.

Commercial data reports are generated using MS EXCEL. All the pertinent client information and the analysis results are entered manually. The draft report is subjected to a 100% technical and completeness review before it is printed out in its final form.

CLP data reports are generated using specialized software (Thru-Put TARGET for organic analyses and Ward Scientific EDR for inorganic analysis). These reports also undergo a 100% review before they are generated in their final form.

Records are maintained for all data, even those results that are rejected as invalid.

#### 11.4.2 Data Reporting for Massachusetts Drinking Water Samples:

Drinking water data reports generated for clients in the State of Massachusetts need to be reported on state forms. These reports are sent to the client. The client is responsible for forwarding copies of the report to the regional DEP Offices and local officials.

#### 11.5 Levels of Data Review:

MITKEM employs five (5) levels of data review. These are based on requirements outlined in several government and other environmental analysis programs including the U. S. Army Corps of Engineers, Air Force Center for Environmental Excellence (AFCEE), Naval Facilities Engineering Service Center (NFESC), HAZWRAP, EPA Contract Laboratory Program (CLP), as well as commercial engineering firm programs.

The data review and evaluation process is structured to insure that all data reported to customers has been thoroughly reviewed and approved using a multistep process designed to identify and correct any error. At any step in the data evaluation and review process, the reviewer has the responsibility and authority to return any data not meeting requirements back to the previous step for re-analysis or correction. No reports are released to the client as final data without successfully passing through each step in the data evaluation and review process. Any data released prior to the completion of the full review process are released with the statement that the data is preliminary pending final review.

The five levels of data review are as follows:

#### 11.5.1 Level 1:

A Level 1 review is performed by the analyst or a qualified peer analyst within the analytical laboratory section that produced the results. Level 1 review is comprehensive, evaluating 100% of the data for compliance with SOP and method requirements, as well as project-specific requirements. The analyst/peer reviews the data set to insure that sample preparation and analysis data are correct and complete. A checklist (Figures 11.5-1, 11.5-2 and 11.5-3) is used to document that Level 1 review has been completed for each data set produced. The specific items reviewed may vary by method, but generally include the items listed below:

- All manual calculations or data entry steps
- Use of proper significant figures and rounding

- That results are compliant with precision and accuracy requirements through evaluation of calibration, blank, LCS, spike, duplicate and/or duplicate spike QC results
- An evaluation of analysis dates in comparison to holding times
- That all analytes are within the calibration range of the test, and any necessary dilutions have been performed.
- That data are complete; that every sample for a work order or sample delivery group that requires this test has been analyzed.
- That spectral identification for target analytes or tentatively identified compounds are correct.
- Spot check computer calculations to insure they are being performed correctly.
- That any deviations from the SOP, method, or project-specific requirements, or any unusual occurrences during analysis are described for inclusion in the report narrative.

#### 11.5.2 Level 2:

Level 2 review is a technical review performed by the supervisor of the analytical laboratory section producing the data, another senior chemist experienced in the particular analysis, or other senior laboratory management, such as the Technical Director, Operations Manager, or QA Director. The same individual may not perform Level 1 and Level 2 review on the same data set. Level 2 review is performed on 100% of the data generated. This review may be less comprehensive that Level 1 review in that it is designed to insure that the Level 1 review was completed for each data set produced. All items listed under Level 1 review above may be checked, with particular focus on the following:

- That all project-specific criteria have been met
- That result flags have been properly applied for any dilutions, calibration failures, blank contamination, etc
- That the results are reasonable when compared to historic or on-going data for this program or for this analysis in general
- Spot checks of manual calculations or data entry steps

- Review the use of significant figures and rounding
- That results are compliant with precision and accuracy requirements through evaluation of performance indicators such as blanks, LCS, surrogate and matrix spikes or duplicate QC results
- Spot check of spectral identifications for target analytes or tentatively identified compounds
- That any notations regarding deviations from SOP, method or project specific requirements, or any unusual occurrences are properly described for inclusion in the report narrative, and to add review comments as necessary.

#### 11.5.3 Level 3:

Level 3 review is an administrative or non-technical review. A level 3 review is evaluated by the report group coordinator, document control specialist, project manager, or other personnel in the data report group. The same person may not both enter the data and review the data entry. 100% of the data manually entered into the commercial data reporting system are reviewed to insure there are no data entry errors. All manual data entry steps used to produce electronic deliverables are also checked.

Data reported using MITKEM's commercial data reporting system are evaluated somewhat differently from those produced using the CLP-type data reporting system, based on the different potential sources of error in these systems. The data review checklist is used to document Level 3 review has been completed on each data set. Additional forms are also used for CLP and CLP-type data assembly and review. The following items are checked during Level 3 review:

- All typographical data entry into commercial data reporting templates
- The client sample identifications are listed correctly for every sample
- The completeness of the data report; that every analysis on the login sheet has been accounted for in the final report
- That results and units are consistent throughout the data set
- That any special requests or other notes on the login sheet have been addressed

 That a description of any flags and data qualifiers is included in the data report.

The review of all sample login and chain of custody information is also included in Level 3 review. The review is evaluated by the project manager immediately following receipt of the samples and production of login paperwork. This review is documented by initialing on the appropriate line on the MITKEM sample login sheet.

#### 11.5.4 Level 4:

Level 4 review consists of the final management approval for the entire data report. Level 4 review is evaluated by senior laboratory management personnel, such as the Technical Director, Operations Manager, QA Director, or Project Manager. This review and sign-off constitutes MITKEM's approval to release the final data report to the client. The signature on the report narrative documents that Level 4 data review has been completed on the entire data report. Level 4 data review consists of:

- That any deviations from method or SOP requirements have been documented and explained such that they will be clear and understandable to the client
- That all unusual occurrences have been clearly described in the report narrative
- That any special analytical requests made by the client have been addressed and adequately recorded in the report
- That the analytical report meets the goals of the testing program
- That the data are reasonable from an overall perspective, for example, that hexavalent chromium does not exceed total chromium, or that dissolved metals do not exceed total metal concentrations.
- That the final report format and appearance are professional and consistent with MITKEM's practice.

#### 11.5.5 Level 5:

The fifth level of data review is performed by the QA/QC Director on a subset of all data produced by the laboratory. QA review is performed on approximately 10% of all data reports generated by the laboratory, with results from each analytical section being represented. Level 5/QA Review usually takes place following release of the data report to the

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client. During Level 5 review, reports are evaluated to check the proper functioning of the entire data acquisition, reduction, evaluation and reporting process. This is accomplished through spot checks and detailed calculation reviews of various steps in the analysis and data reporting process. The specific items checked are at the discretion of the QA/QC Director. Level 5 review functions as an additional check that the laboratory's QA systems are operating properly. Any deficiencies encountered during Level 5 reviews are promptly reported to MITKEM \* senior management.

Flow charts of the data review process follow in Figure 11.5-4.

#### 11.6 Document Control:

All login sheets, Chains-of-Custody (COC) and Sample Condition Forms (SCF) are generated in Sample Receiving. Samples are signed in/out of the sample receiving area by analysts. In the Prep lab, samples and all pertinent information is logged into the logbooks. Once samples are transferred to the instrument lab, the extracts are written in the transfer logbook. In the instrument lab, the extracts are recorded in the instrument run log. All QC of ICAL, CAL and raw data are kept in the instrument lab. Results go into the project file in data reporting. The data is reviewed. The original copy is sent to the client. A copy of the results is kept in the project file. The project files are kept onsite in a storage area for 6 months. The files are then shipped to an offsite storage area where they will remain for an additional 6 ½ years. After this time, the files will be destroyed: All controlled documents including SOPs, QA Manuals, Logbooks, etc. are dated. This is the date the document is controlled and will stay in force until the next official/controlled update.

#### 11.6.1 Logbooks:

All logbooks are issued and controlled by the QA Department. When logbooks are complete, the QA Department archives them in order of control number for a minimum of ten (10) years. The logbooks are stored in an on-site storage facility for a minimum of 6 months and then boxed and stored in a locked off-site storage facility.

#### 11.6.2 Project/Data Files:

MITKEM is a secured, limited access building. The doors are secured with a keypad entry system. All information pertaining to the analysis of samples is maintained and stored in a project file folder. This information includes all login sheets, COC, SCF, bench sheets and analytical data. File folders containing all hard copy data and chain-of-custody records are stored in an off-site storage facility for an additional 6 ½ years. After 7

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years, files are shredded. The off-site storage facility is a locked storage area. Access is limited to the CFO and request will be made to the CFO.

In the event Mitkem Corporation changes ownership, the maintenance, control, storage and eventual disposal at the end of the appropriate time period, of all records, including client data and QA/QC files, will transfer to the new owners.

In the event Mitkem Corporation decides to cease operations, clients will be notified prior to the cessation of operations and their files/records will be made available to them. Within a designated time period after notification, the client will be responsible for taking custody and the future maintenance of their records. If the client determines they do not want to maintain the records, these will be disposed of properly.

### 11.6.3 Standard Operating Procedures (SOPs):

SOPs are prepared by the Lab Supervisor in conjunction with the QA/QC Director, reviewed and approved by the Lab Supervisor/Manager, Operations Manager and QA/QC Director and distributed as controlled documents by the QA/QC Director. All SOPs are reviewed and updated as necessary on an annual basis. The procedure for preparing, reviewing, approving, revising and distributing SOPs are described in SOP No.O01.

#### 11.6 4 Method Updates:

It is the laboratory's policy to implement new revisions of frequently used methods within six months of the date the method revision is promulgated. The QA/QC Director and Technical Director make the final decision on when a method revision will be adopted by the laboratory. When the laboratory is in the middle of a client's sampling project, the lab will continue using the same revision for the entire sampling event unless advised otherwise by the client. Consequently, once the laboratory has formally adopted a new method revision, both the old and new revision may be in use at the same time, depending on the project.

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## Figure 11.5-1 Commercial Data Review Checklist

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## Figure 11.5-1 Commercial Data Review Checklist

## Mitkem Corporation

## Data Review Checklist

Decient Number	Analysis:	Matrix
Project Number:		-
Level 1 Review:	Level 2 Review:	Level 3 Review:
7	es No –List/Explain any Un	nusual Occurrences or Nonconformances
		•
Calibration Acceptable Tune/ICIL/CCIL	List all non-conforming project analyt	es .
LCS Acceptable	List all non-conforming project analys	- · ·
Blank Acceptable	List all non-conforming project analyte	<u> </u>
Spike Acceptable		
opine recopusio	Reasonable recovery / Matrix effect / S	pike to sample concentration ratio
Dup / MSD Acceptable	Reasonable procision / Sample non-ho	mogencity?
Within Holding Time	List runs/re-runs oud of holding time; l	
Within Instrument Range	Dilutions properly noted: Explain any	"E" flag analytes or dilutions with no target hit
Surrogates Acceptable		The state of the s
Yandan Dadama	List all non-conforming analytes, Matr	tr effect?
Identification Reviewed	Potential for false positives checked?	
Calculation Check:	Including proper significant figures, re	ounding
Reasonableness Check:	Compared to historic or on-going trends	s, or for this analysis in general?
Typographical Review:	Notes:	×
Client ID Check:		
Completeness Check:	_	•
Consistency Check:		<b>,</b>
Special Requests:		

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## Figure 11.5-2 CLP and CLP-type Data Review Checklist – Organics

## Figure 11.5-2 CLP and CLP-type Data Review Checklist – Organics

Date Initiated 1/14/99

# Mitkem Corporation CLP/CLP-like Deliverable Review Check List for Organics Analysis

Project Number:	An	ıalysis:	(1.00	_ Fraction	Fraction:							
Target:		tegory:	(ASP onl	<u>(y)</u> Analy: Correc	y) Analyst: Correction by Analyst:							
Data Pack. Assembly:	Da	na rack. No	vicw	_ 000								
		<u>Analyst</u>	Date:	. 1	Reviewer	Date:						
SDG Summary Sheet Alkane Summary Sheet Sample Log-in Sheet Extraction Bench Sheet % Solid Bench Sheet Extract Transfer Log Instrument Run Log GPC Run Log	Pages		sual Observation	Check		Comments						
Internal Sample Tracking Log												
	Client IDs	OK/Unu	sual Observation	Check	<u>C</u>	<u>omments</u>						
Holding Time												
Surrogate												
Initial Analysis at Dilution				<del></del>								
"RE" Samples						······································						
"DL" Samples							<del>-</del>					
MS/MSD Samples												
	Sample /Set #	<u>OK/Unu</u>	sual Observation	<u>Check</u>	<u>C</u>	omments						
Blank .			•									
Las												
Tune												
Initial Calibration												
Continuing Calibration												
Internal Standard Area							<u></u>					
Note:				Client II ID Trund Special I	cation	<u>Yස</u>	<u>No</u>					

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# Figure 11.5-3 CLP and CLP-type Data Review Checklist – Inorganics

# Figure 11.5-3 CLP and CLP-type Data Review Checklist – Inorganics

## MITKEM CORPORATION

### CLP/CLP-like Deliverable Check List for Inorganic Analysis

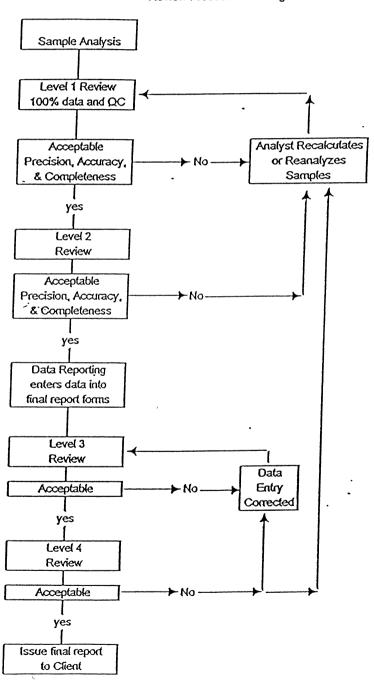
Pro	ject N	umt	er:_												An:	alysi	s:							
Project Number:P.C. #:											Category: (ASP only							(X)						
Input by/date							_					Reviewer:						_						
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					•						•													
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Prep	Log	She	et (A	Q/S	L)					-														
% S	olid B	end	h Sh	eet						-												·		
Tum	bling	Log	(ТС	LP/S	SPLF	?)				-											·			
							2	Chec	k		Lab	JD			Ω	K/Ur	บรบ	al Ot	sery	ation	/Dev	iation	1/Elag	IS.
Dilut	ed Sa	mpl	es											-	-									
Spik	ed Sa	mpl	es (f	٧)										-										
Dupi	icate :	Sam	ples	(*)										_	_				-		•			<del></del>
Seria	al Dilu	tion:	s (E)	)										-	-	•						<b>-</b>	<del></del>	<del>-</del>
Blani	ks														-							··		<del></del>
LCS														-	-							•••	-	
ICP I	Interfe	ren	се											•	-									<del></del>
Dran	/Anai	lve	s Na	dac.																	Ye	S		No
iteh	n Pu Idi	iyət	2 INC	NGS.	•									(	Slien	t ID	Che	ck:					-	
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										5	Special Request:					_								

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Figure 11.5-4 Data Review Flow Diagram

Figure 11.5-4
Data Review Flow Diagram

#### MITKEM CORPORATION Review Process Flow Diagram



#### 12.0 LABORATORY QUALITY CONTROL CHECKS

MITKEM analytical and field procedures are based on sound quality control methodology, which derives from three primary sources:

- 1. Standards for Good Laboratory Practice,
- 2. Specific EPA and other approved analytical methods, and
- 3. "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA 600/4-79-019).

In the application of established analytical procedures, MITKEM employs, at a minimum, the QC protocols described in the references found in the Analytical Methods section of this document. Specific projects may require additional quality control measures, due to such factors as difficult sample matrices or use of innovative techniques. For those projects MITKEM will recommend and implement, subject to client approval, the QC measures to produce data of known quality.

Each of the MITKEM laboratory departments have an individual QC program, which includes, but is not limited to, the practices described below.

#### 12.1 Detection Limit Determination/Verification:

Detection Limits are developed annually for all inorganic and organic target compounds.

#### 12.2 Personnel Training:

Chemists who begin their employment at MITKEM are first instructed under the MITKEM Safety Training Program. Before performing and analyses, a chemist is required to read the appropriate protocols and SOPs. He/she must become familiar with the laboratory equipment and the analytical methods. The chemists begin a training period during which they work under strict supervision. Independent work is only permitted after the chemist successfully completes a proficiency review. Copies of results, if any, of training sessions and training course documentation will be placed in the employee's training file archived by the QA Director.

#### 12.3 Control Charts:

For organic and inorganic analyses, the recoveries of analytes in the lab control samples are plotted on control charts. These charts are used to establish control and warning limits.

12.3.1 Control limits are calculated and updated at least annually from the LCS data points for each analyte and matrix using the following equations:

$$Average(\bar{x}) = \frac{\left[\sum_{i=1}^{n} x_{i}\right]}{n}$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \overline{x})^2}{n-1}}$$

where:

SD = Standard Deviation N = number of data points

Warning Limits = Average  $\pm 2 * SD$ 

Control Limits = Average  $\pm 3 * SD$ 

- 12.3.2 Control limits must be approved by the QA/QC Director and by the Technical Director prior to adoption by the laboratory. In the event that limits are wider than method recommended limits, the method recommended limits may be adopted and the analytical procedure will be re-evaluated to determine possible causes. Additionally, in the event that control limits are tighter than 10% from the average, the lab may adopt a control limit of ±10% from the average.
- 12.3.3 Control charts are plotted in EXCEL using the template in the QA Department directory:/Control Charts/Control Chart Template.xls.

Data from each laboratory is downloaded into an EXCEL spreadsheet. The compounds, recoveries, and date analyzed for each file is copied into page one of the Control Chart Template. The data for each analyte is then

automatically read into another page of the spreadsheet (one page for each analyte) and plotted onto a control chart. Control charts are generated for each analyte in the inorganic section, and for a representative sampling of analytes in the organic sections. Each control chart is then printed for review by the QA/QC Director and by the Lab Supervisor/Manager. Out of control situations noted on the control chart are brought to the attention of the Technical Director by the QA/QC Director.

An example control chart is presented as Figure 12.3-1. LCS data must be reviewed and evaluated daily against the Control Limits to establish that the system is in control.

- 12.3.4 The following situations constitute an out of control situation on a control chart:
  - One data point above or below the Control Limit line.
  - Two consecutive data points above or below the Warning Limit line.
  - Six or more consecutive data points above the Average Line or six or more consecutive data points below the Average Line. This situation suggests a trend and suggests the procedure has been changed in some way (for better or worse). The cause for this trend must be investigated.

# 12.4 General QC Protocols:

Organics Laboratory;

- Trip blanks and holding blanks, when applicable, are analyzed to detect contamination during sample shipping, handling and storage.
- Method blanks, at a minimum of one in every 20 samples, are analyzed to detect contamination during analysis.
- Volatile organic method blanks are analyzed once during each analytical sequence.
- One blank spike of an analytical sample or laboratory water or Ottawa sand every 20 samples, is analyzed to determine accuracy.
- Sample spikes and spike duplicates, as requisitioned, are analyzed to determine accuracy and the presence of matrix effects. The Relative Percent Difference (RPD) is also determined for matrix spike/matrix spike duplicates. The criteria followed is stated in the individual methods.

- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA QA/QC standards.
- Surrogate standards are added to samples and calculations of surrogate recoveries are performed to determine matrix effect.
- Internal standards for GC/MS analysis are added to sample extracts to account for sample-to-sample variation.
- GC analysis of EPA traceable standards to verify working standard accuracy and instrument performance.
- Initial multi-level calibrations are performed to establish calibration curves.
- Instruments are calibrated with every analytical sequence.
- Tuning of GC/MS systems once every 12 hours to EPA specifications is implemented for consistency in data generation.

When QC limits are not met during an analytical run, those samples affected must be re-analyzed after the instrument has been re-calibrated. If QC limits continue to be out of control, the instrument must be checked and/or a service call made.

## Inorganic Laboratory;

- Trip blanks are analyzed when applicable, to detect contamination during sample shipping, handling and storage.
- Method blanks are analyzed at a minimum of one every 20 samples, to detect contamination during analysis.
- One matrix spike and matrix spike duplicate of an analytical sample or laboratory water or soil is made and spike recoveries are computed every 20 samples to determine accuracy. If insufficient volume of sample is received, one duplicate sample is analyzed at a frequency of one per batch or 20 samples.
- Performance evaluation samples from EPA and state agencies are analyzed to verify continuing compliance with EPA QA/QC standards.
- Instruments are calibrated daily.
- QC checks samples are analyzed during every analytical run to document accuracy.

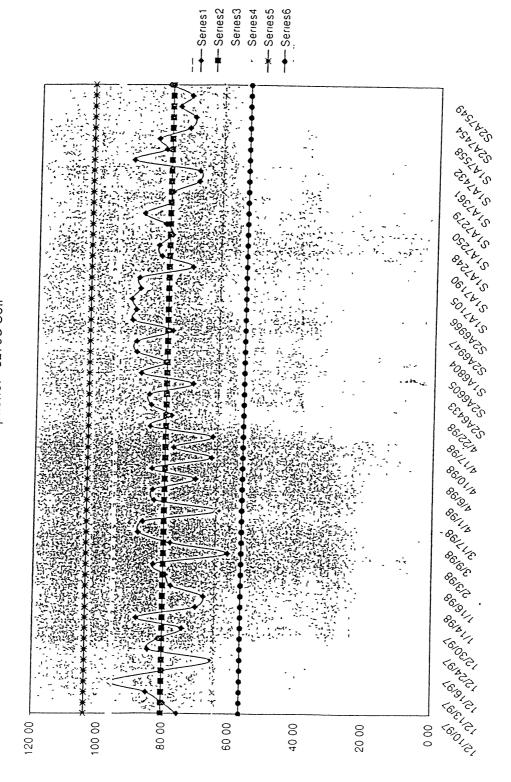
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When QC limits are not met during an analytical run, those samples affected must be re-analyzed after the instrument has been re-calibrated. If QC limits continue to be out of control, the instrument must be checked and/or service call made.

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Figure 12.3-1
Example Control Chart

Figure 12.3-1 Example Control Chart



Nitrophenol - 8270C Soi

# 13.0 QUALITY ASSURANCE SYSTEMS AUDITS, PERFORMANCE AUDITS AND FREQUENCIES

The MITKEM Quality Assurance staff performs routine internal audits of the laboratory. The frequency of such audits depends on the workload in house but is done quarterly, at a minimum. These audits entail reviewing laboratory note books and all appropriate operations to ensure that all laboratory systems including sample control, analytical procedures, data generation and documentation meet contractual requirements and comply with good laboratory practices.

# 13.1 System Audits:

The laboratory is audited quarterly by the QA/QC Director in order to detect any sample flow, analytical or documentation problems and to ensure adherence to good laboratory practices as described in MITKEM standard operating procedures and quality assurance plan. The checklist used in an internal systems audit at MITKEM is presented in Figure 13.1-1. Problem areas detected during the annual Systems Audit is monitored monthly in follow-up audits conducted by the QA staff.

### 13.2 Performance Audits:

MITKEM participates quarterly in external Performance Evaluation (PE) studies including the Water Supply (WS) and Water Pollution (WP) Series of PEs administered by the Quality Assurance Branch of the EPA.

Internally, performance is monitored on a daily basis at MITKEM through the use of surrogate standards, LCSW and LCSS samples. Check samples from independent commercial sources are employed routinely in each of the MITKEM laboratory departments and ensure continuing high level performance. Internal blind PE samples are distributed to each laboratory department by the QA/QC Director at a minimal frequency of semiannually.

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Figure 13.1-1 QA Systems Audit Checklist

# MITKEM CORPORATION QA Internal Audit Aug-99

# I. Quality Assurance

QA/QC Director with assigned duties?	Yes / No
QA Report to Management submitted monthly?	Yes / No
Organizational Chart Up to Date? (Attachment A)	Yes / No
Quality Assurance Plan Updated Annually?	Yes / No
Date Revised:	
Is the Quality Assurance Plan a controlled document?	Yes / No
Laboratory Equipment	
Is equipment adequate and up to date?	Yes / No
Attach current Equipment List (Attachment B)	
Audit Program	
Internal Systems Audits performed annually?	Yes / No
Attach listof External Systems Audits from last year.	
(Attachment C)	
Internal Performance Audits performed quarterly?	Yes / No
Attach list of External Performance Audits from last year	
(Attachment D)	37 / 37
Internal Data Audits performed on 10% of data generated?	Yes / No
Employee Training	
Employee Training Files up to date?	Yes / No
Safety Training Record for all employees?	Yes / No
Attach current list of employees and job titles. (Attachment E)	
Standard Operating Procedures	
Are SOPs updated annually for each analytical method?	Yes / No
Are SOPs updated annually for Sample Receiving?	Yes / No
Are SOPs updated annually for QA/QC Procedures?	Yes / No
Are SOPs updated annually for Data Reporting/Data Review?	Yes / No
Are SOPs updated annually for Standard Traceability?	Yes / No
Are SOPs controlled documents?	Yes / No
Are SOPs signed by appropriate individuals?	Yes / No
Method Validation	
Initial Demonstration of Proficiency before method is implemented?	Yes / No

	Are MDL studies up to date for each method?	Yes / No
	Is the Amount Spiked equal to 3-5x the calculated MDL?	Yes / No
	Attach MDL study schedule (Attachment F)	
	Does the lab maintain a copy of each method it performs?	Yes / No
	Corrective Actions	
	Is a formal system for Corrective Actions in place?	Yes / No
	Does the QA/QC Director review CARs?	Yes / No
	Are CARs controlled documents?	Yes / No
	<u>Logbooks</u>	
	Are laboratory logbooks controlled and archived by QA?	Yes / No
	Are logbook templates controlled and archived by QA?	Yes / No
	Are logbooks peer reviewed monthly?	Yes / No
	Proper correction techniques used?	Yes / No
	Empty spaces properly "z"'d out?	Yes / No
	Are logbooks paginated?	Yes / No
II. (	Quality Control	
	Is an NIST traceable thermometer available?	Yes / No
	Are lab thermometers calibrated annually against the NIST thermometer?	Yes / No
	Are correction factors in use on lab thermometers?	Yes / No
	Are Class "S" weights calibrated NIST traceable annually?	Yes / No
	Are balances serviced annually?	Yes / No
	Are balances calibrated monthly and calibration recorded?	Yes / No
	Is balance calibration acceptance criteria clearly defined and posted?	Yes / No
	Control Charts	
	Are control charts in place for each method and matrix?	Yes / No
	Does each chart have a minimum of 30 points?	Yes / No
	Are control charts updated monthly?	Yes / No
	Are control limits updated annually or when major method changes are	
	made?	Yes / No
	Are control limits issued to labs as controlled documents?	Yes / No
	Standard Traceability/Equivalency	
	Are standards labelled with standard name, concentration, solvent,	
	working standard ID, expiration date, and preparer's initials?	Yes / No
	Are expiration dates of standards clearly defined in an SOP?	Yes / No
	Are standards QC's against a second source standard after each initial	** /*-
	calibration?	Yes / No

Are standards traceable from working standard analysis back to the	
standard received date, manufacturer, and lot#?	Yes / No
Are solvents traceable from preparation logbook to date received,	
manufacturer, and lot#?	Yes / No
III. Sample Receiving	
Is an up to date SOP present in the area?	Yes / No
Is a sample receiving checklist used to receive samples?	Yes / No
Condition of samples on receipt?	Yes / No
Sample temperature on receipt?	Yes / No
Radiation screen?	Yes / No
C-O-C signed and properly filled out?	Yes / No
Sample Storage	
Are samples, except aqueous metals, refrigerated at $4^{\circ} \pm 2^{\circ}$ C?	Yes / No
Are refrigerator temperatures checked daily?	Yes / No
Are aqueous metals stored at room temperature?	Yes / No
Is sample pH checked and recorded for samples requiring acid/base	
preservation?	Yes / No
Are high concentration VOAs stored separately from other samples?	Yes / No
Are VOA samples stored separately from other samples?	Yes / No
Is a system of corrective actions in place?	Yes / No
Sample Containers	
Are sampling instructions provided with sample containers?	Yes / No
Are proper preservations, sample containers, etc. posted?	Yes / No
Are preservatives traceable to original manufacturer & lot?	Yes / No
Are containers precleaned by the manufacturer and a certificate of	
cleanliness supplied?	Yes / No
Sample Log-In	
Is a unique ID assigned to each sample?	Yes / No
Is each sample container uniquely identified?	Yes / No
Is there a peer review of sample labelling procedures?	Yes / No
Waste Disposal	
Do internal COC procedures exist from receipt to disposal?	Yes / No
Are samples disposed by a company certified to dispose of hazardous	
waste?	Yes / No
Is a certificate of disposal received and filed?	Yes / No

Safety	
Are safety glasses, lab coat, and gloves worn by the sample custod	lian? Yes / No
Are sample coolers opened under a ventilated hood?	Yes / No
IV. Data Reporting/Data Review	
Has the Data Review SOP been reviewed/updated annually?	Yes / No
Are Data Reviews clearly documented with the use of checklists?	Yes / No
Is 100% of data peer reviewed?	Yes / No
Is data reviewed technically by a Lab Supervisor/Lab Manager?	Yes / No
Is 10% of data reviewed by the QA/QC Officer?	Yes / No
Are estimated concentrations reported for values found between	
the Reporting Limit and Method Detection Limit? (USA	ACE) Yes / No
Is a system in place for archiving data reports?	Yes / No
How long are data reports kept?	
V. Inorganics	
Logbooks	~~ (>7
Does a run logbook exist for each analytical instrument?	Yes / No
Does an instrument maintenance log exist for each instrument?	Yes / No
Does a prep log exist for each procedure?	Yes / No
Are logbooks peer reviewed monthly?	Yes / No
Proper correction techniques?	Yes / No
Empty spaces "z"'d out?	Yes / No
Paginated?	Yes / No
Controlled?	Yes / No
Do logbooks contain all pertinent information to the procedure?  ( i.e., method, matrix, reagent lot#, digestion temp., etc	Yes / No .)
	,
Standards Are standards QC'd against a second source after each ICAL?	Yes / No
Are standards traceable throughout the lab?	Yes / No
Are expired standards present in the lab?	Yes / No
Is there a defined system for assigning expiration dates?	Yes / No
Analytical Methods	
Are SOPs method compliant?	Yes / No
Do analysts follow the SOP?	Yes / No
Do analysts do an initial demonstration of proficiency study?	Yes / No
Are analysts adequately trained and knowledgeable?	Yes / No
Does the IEC contain all analytes that interfere with target analyte	tes? Yes / No

(not just Ca, Fe, Al, Mg)	
Is ICAL documentation maintained on file in the lab?	Yes / No
Corrective Actions	
Is there a system for corrective actions in place?	Yes / No
<u>Safety</u>	
Do analysts wear safety glasses, lab coats, and gloves?	Yes / No
Are all reagents handled under a hood?	Yes / No
VI. Volatiles	
<u>Logbooks</u>	
Does a run logbook exist for each analytical instrument?	Yes / No
Does an instrument maintenance log exist for each instrument?	
Are logbooks peer reviewed monthly?	Yes / No
Proper correction techniques?	Yes / No
Empty spaces "z"'d out?	Yes / No
Paginated?	Yes / No
Controlled?	Yes / No
Do logbooks contain all pertinent information to the procedure (i.e., method, matrix, reagent lot#, soil weight, etc.)	? Yes / No
<u>Standards</u>	
Are standards QC'd against a second source after each ICAL?	Yes / No
Are standards traceable throughout the lab?	Yes / No
Are expired standards present in the lab?	Yes / No
Is there a defined system for assigning expiration dates?	Yes / No
Is standard freezer temperature monitored?	
Analytical Methods	
Are SOPs method compliant?	Yes / No
Do analysts follow the SOP?	Yes / No
Do analysts do an initial demonstration of proficiency study?	Yes / No
Are analysts adequately trained and knowledgeable?	Yes / No
Is ICAL documentation maintained on file in the lab?	Yes / No
When %RSD > 15%, is linear regression curve fit adopted?	Yes / No
(linear regression criteria =)	
Is a CCV run at the end of the analytical sequence? (USACE)	Yes / No
Corrective Actions	
Is there a system for corrective actions in place?	Yes / No

٠.

	Safety Are all reagents handled under a hood?	Yes / No
VII.	Semivolatiles	
	<u>Logbooks</u>	
	Does a run logbook exist for each analytical instrument?	Yes / No
	Does an instrument maintenance log exist for each instrument?	Yes / No
	Are logbooks peer reviewed monthly?	Yes / No
	Proper correction techniques?	Yes 'No
	Empty spaces "z"'d out?	Yes No
	Paginated?	Yes / No
	Controlled?	Yes / No
	Do logbooks contain all pertinent information to the procedure?  ( i.e., method, matrix, reagent lot#, etc.)	Yes / No
	<u>Standards</u>	
	Are standards QC'd against a second source after each ICAL?	Yes / No
	Are standards traceable throughout the lab?	Yes / No
	Are expired standards present in the lab?	Yes / No
	Is there a defined system for assigning expiration dates?	Yes / No
	Is standard frèezer temperature monitored?	Yes / No
	Analytical Methods	** />*
	Are SOPs method compliant?	Yes / No
	Do analysts follow the SOP?	Yes / No
	Do analysts do an initial demonstration of proficiency study?	Yes / No
	Are analysts adequately trained and knowledgeable?	Yes / No
	Is ICAL documentation maintained on file in the lab?	Yes / No
	When %RSD > 15%, is linear regression curve fit adopted?  (linear regression criteria =)	Yes / No
	Is a CCV run at the end of the analytical sequence? (USACE)	Yes / No
	Is a Method Blank analyzed after each CCV?	Yes / No
	Is DDT breakdown and tailing factors for benzidine and pentachlorophenol	2 33 7 2 13
	evaluated for acceptability?	Yes / No
	Does analyst review data for false negatives?	Yes / No
	Corrective Actions	
	Is there a system for corrective actions in place?	Yes / No
	Safety	

	Are all reagents handled under a hood?	Yes / No
VIII.	Pesticides/PCBs	
	Logbooks	
	Does a run logbook exist for each analytical instrument?	Yes 'No
	Does an instrument maintenance log exist for each instrument?	Yes No
	Are logbooks peer reviewed monthly?	Yes No
	Proper correction techniques?	Yes No
	Empty spaces "z"'d out?	Yes No
	Paginated?	Yes No
	Controlled?	Yes No
	Do logbooks contain all pertinent information to the procedure?  ( i.e., method, matrix, reagent lot#, etc.)	Yes No
	<u>Standards</u>	
	Are standards QC'd against a second source after each ICAL?	Yes / No
	Are standards traceable throughout the lab?	Yes / No
	Are expired standards present in the lab?	Yes / No
	Is there a defined system for assigning expiration dates?	Yes / No
	Is standard freezer temperature monitored?	Yes / No
	Analytical Methods	
	Are SOPs method compliant?	Yes / No
	Do analysts follow the SOP?	Yes / No
	Do analysts do an initial demonstration of proficiency study?	Yes / No
	Are analysts adequately trained and knowledgeable?	Yes / No
	Is ICAL documentation maintained on file in the lab?	Yes / No
	When %RSD > 15%, is linear regression curve fit adopted?	Yes / No
	(linear regression criteria =)	
	Are quadratic fits used excessively in ICALs?	Yes / No
	Is a CCV run after every 10 samples? (USACE)	Yes / No
	Is a Method Blank analyzed after each CCV?	Yes / No
	Is DDT & Endrin breakdown monitored for PCB only analyses?	Yes / No
	Are QC samples run on same instrument as field samples?	Yes / No
	Are retention time studies performed after each column change?	Yes / No
	Is target analyte %D between primary and confirmation <40%?	Yes / No
	Corrective Actions	
	Is there a system for corrective actions in place?	Yes / No
	Safety	

Are all reagents handled under a hood?	Yes / No
VIX. Organic Preparation	
Logbooks	
Does a preparation logbook exist?	Yes / No
Does a run logbook exist for each instrument?	Yes No
Does an instrument maintenance log exist for each instrument?	Yes No
Are logbooks peer reviewed monthly?	Yes / No
Proper correction techniques?	Yes No
Empty spaces "z"'d out?	Yes No
Paginated?	Yes No
Controlled?	Yes No
Do logbooks contain all pertinent information to the procedure? (i.e., method, matrix, reagent lot#, pH, % solids, etc.)	Yes No
<u>Standards</u>	
Are standards QC'd against a second source after each ICAL?	Yes / No
Are standards traceable throughout the lab?	Yes / No
Are expired standards present in the lab?	Yes No
Is there a defined system for assigning expiration dates?	Yes / No
Is standard freezer temperature monitored?	Yes / No
Are solvents traceable through preparation?	Yes / No
Are personnel aware of syringe tolerances?	Yes / No
Analytical Methods	/37
Are SOPs method compliant?	Yes / No
Do analysts follow the SOP?	Yes / No
Do analysts do an initial demonstration of proficiency study?	Yes / No
Are analysts adequately trained and knowledgeable?	Yes / No
Is ICAL documentation maintained on file in the lab?	Yes / No
Are temperatures of water baths and hot plates monitored?	Yes / No
Is deionized, charcoal -filtered water used for Pest/PCB blanks?	Yes / No
Corrective Actions	
Is there a system for corrective actions in place?	Yes / No
Safety	
Do analysts wear safety glasses and lab coat?	Yes / No
Are all reagents handled under a hood?	Yes / No
Comments	

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### 14.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a routine practice at MITKEM for all instrumentation. Scheduled preventive maintenance minimizes instrument downtime and subsequent interruption of analysis. All major instrumentation are under service contracts so that downtime due to catastrophic events are minimized.

Only those equipment items meeting or exceeding applicable performance requirements are used for data collection. This includes items such as laboratory balances as well as major analytical instruments such as ICPs, GCs and GC/MSs.

MITKEM's laboratory personnel are familiar with the routine and non-routine maintenance requirements of the instruments they operate. This familiarity is based on education, hands-on experience and manufacturer's training courses.

### **GC** Maintenance:

- 1. The injection septum will be replaced once every fifty (50) injections or earlier if a leak develops.
- 2. The injection liner will be replaced once every fifty (50) injections or when initial and/or continuing calibrations fails repeatedly to meet method requirements.
- 3. The column will be replaced if chromatograms show excessive peak tailing and/or initial and continuous calibration verifications fail repeatedly to meet method requirements.
- 4. Once a year, under service contract, all GC equipment undergo extensive maintenance by a manufacturer's service engineer.

## GC/MS Maintenance:

- 1. GC injector and liner are cleaned daily.
- 2. The column will be replaced if chromatograms show excessive peak tailing and/or initial and continuous calibration verifications fail repeatedly to meet method requirements.
- 3. The ion source will be cleaned when initial and/or continuing calibration repeatedly fail method specified criteria.
- 4. The pump oil will be replaced once a year.
- 5. Once a year, under service contract, all GC/MS systems undergo extensive maintenance by a manufacturer's service engineer.

#### **ICAP** Maintenance:

- 1. Peristaltic pump tubing will be replaced every sixteen (16) hours of instrument time or sooner when memory effects are manifested.
- 2. The plasma torch is cleaned with (aqua regia) at least once a week. If memory effect are manifested the torch will be cleaned immediately.
- 3. The sample introduction (spray chamber and nebulizer) is cleaned at least once a week.
- 4. Air filters are cleaned once every two (2) weeks or as needed upon visual inspection.
- 5. Once every six (6) months, under service contract, the instrument undergoes extensive maintenance by a manufacturer's service engineer

### **GFAA** Maintenance

- 1. The quartz windows are cleaned before every analysis.
- 2. Furnace tubes are replaced every 250 burns or when needed upon visual inspection.
- 3. Once every six (6) months, under service contract, the instrument undergoes extensive maintenance by a manufacturer's service engineer.

## Mercury FIAS 100 Maintenance

- 1. Pump tubing is replaced every 48 hours of instrument run time.
- 2. The windows of the optical cell are cleaned before each analysis.
- 3. The inside of the optical cell is cleaned once every 48 hours of instrument run time.
- 4. Once every six (6) months, under service contract, the instrument undergoes extensive maintenance by a manufacturer's service engineer.

# Lachat 8000 Maintenance

- 1. All pump tubing is replaced every 48 hours of instrument run time.
- 2. Auto sampler arm is lubricated every 48 hours of instrument run time.

3. Once every six (6) months, under service contract, the instrument undergoes extensive maintenance by a manufacturer's service engineer.

### TCLP/SPLP Tumbler Maintenance

- 1. The tumbler is checked every week for number of rotations per minute (30rpms)
- 2. If the tumbler is not spinning at 30rpms, motor is cleaned and oiled.
- 3. If tumbler is not spinning at 30rpms after maintenance, the motor will be replaced.

Instrument maintenance logs are kept for each instrument (Figure 14-1). The person performing the maintenance is required to provide the following information in the log:

- Equipment identifier
- The inspection, maintenance, calibration or corrective action(s) performed.
- The trigger(s) for the maintenance action(s)
- The identity of the person(s) performing the maintenance
- The date on which the work was performed, and
- The condition of the equipment upon completion of the work.

MITKEM maintains an inventory of replacement parts required for preventive maintenance and spare parts that often need replacement, such as filaments for GC/MS systems and the more mundane electrical fuses and GC column ferrules. To control cost, the appropriate supervisor shall decide the types and numbers of spare parts kept on hand for each equipment item.

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# Figure 14-1 Example Instrument Maintenance Log

# Figure 14-1 Example Instrument Maintenance Log

ITKE	EM CORPORATION INSTRUMENT MAINTENA	NCE LOG
	Instrument :	
Date	Maintenance Description .	
Date		
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		`
		•

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# Figure 14-2 Instrument Maintenance Schedule

# Figure 14-2, Page 1 of 2

# MITKEM CORPORATION Preventive Maintenance Schedule

Instrument	Activity	Frequency
Gas Chromatograph (GC)	Injection septum replaced Injection liner replaced	Every 50 injections Every 50 injections
	The column will be replaced if chromatograms show excessive peak tailing and/or initial and continuing calibration verifications fail repeatedly to meet method requirements.  All GC equipment undergo extensive maintenance by the manufacturer's service	As needed
	lengineer.	Annually
GC/MS	GC injector and liner cleaned	Daily
	The column will be replaced if chromatograms show excessive peak tailing and/or initial and continuing calibration verifications fail repeatedly to meet method requirements.  The ion source will be cleaned when initial and/or continuing calibration repeatedly fail	As needed
	method specified criteria.  The pump oil is replaced.	As needed Annually
	All GC/MS systems undergo extensive maintenance by a manufacturer's service engineer.	Annually
Inductively Coupled		Every 16 hours of
Plasma (ICP)	Peristaltic pump tubing is replaced	instrument run time
	The plasma torch is cleaned (aqua regia).  The sample introduction (spray chamber and nebulizer) is cleaned	Weekly Weekly
	Air filters are cleaned.	Biweekly
	The instrument undergoes extensive maintenance by the manufacturer's service engineer.	Semiannually
GFAA	Quartz windows are cleaned	Prior to each analysis
	Furnace tubes are replaced	Every 250 burns
	The instrument undergoes extensive maintenance by the manufacturer's service engineer.	Semiannually

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# Figure 14-2, Page 2 of 2 MITKEM CORPORATION Preventive Maintenance Schedule

Instrument	Activity	Frequency
Mercury FIAS 100	Pump tubing is replaced Windows of the optical cell are cleaned Inside of optical cell is cleaned	Every 48 hours of instrument run time Prior to each analysis Every 48 hours of instrument run time
	The instrument undergoes extensive maintenance by the manufacturer's service engineer.	Semiannually
Lachat 8000	All pump tubing is replaced  Autosampler arm is lubricated	Every 48 hours of instrument run time Every 48 hours of instrument run time
	The instrument undergoes extensive maintenance by the manufacturer's service engineer.	Semiannually
TCLP/SPLP Tumbler	Number of rotation per minute (rpm) checked	Weekly
	Tumbler maintenance when not spinning 30 rpms	Motor cleaned and oiled
	Tumbler is not tumbling 30rpms after maintenance	Motor will be replaced

# 15.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, COMPLETENESS, METHODS DETECTION LIMIT AND LINEAR DYNAMIC RANGE

These mathematical equations represent the means of calculating analytical figures of merit on a routine basis at MITKEM. However, they may be supplanted with other calculations if requested by the client. Precision, accuracy and completeness are also discussed in Section 6.

### 15.1 Precision:

Precision is frequently determined by the comparison of replicates, where replicates result from an original sample that has been split for identical analyses. Standard deviations, s, of a sample are commonly used in estimating precision.

Sample standard deviation, s:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (x_i - \bar{x})^2}$$

where a quantity,  $x_i$  (e.g. a concentration), is measured n times with a mean,  $\bar{x}$ .

The relative standard deviation, RSD (or sample coefficient of variation, CV), which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates (although it may be applied in the case of n = 2).

$$%RSD = 100 (s / \bar{x})$$

or

$$CV = 100 (s / \bar{x})$$

where: RSD = relative standard deviation, or CV = coefficient of variation s = standard deviation  $\bar{x}$  = mean

For duplicates (samples that result when an original sample have been split into two for identical analyses), the relative percent difference (*RPD*) between the two samples may be used to estimate precision.

$$RPD = \frac{2(D_1 - D_2)}{(D_1 + D_2)} \times 100\%$$

where:  $D_I$  = first sample value

 $D_2$  = second sample value (duplicate)

# 15.2 Accuracy:

The determination of accuracy of a measurement requires a knowledge of the true or accepted value for the signal being measured. Accuracy may be calculated in terms of bias as follows:

$$Bias = X - T$$

$$\%Bias = 100 \frac{(X-T)}{T}$$

where: X = average observed value of measurement T = "true" value

Accuracy also may be calculated in terms of the recoveries of analytes in spiked samples:

$$\% \operatorname{Re} \operatorname{cov} \operatorname{ery}(\% R) = 100 \times \frac{\left(SSR - SR\right)}{SA}$$

where: SSR = spikes sample result

SR = sample result SA = spike added

### 15.3 Completeness:

Determine whether a database is complete or incomplete may be quite difficult. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol. Less obvious is whether the data are sufficient to achieve the goals of the project. All data are reviewed in terms of goals in order to determine if the data set is sufficient.

Where possible, the percent completeness for each set of samples is calculated as follows:

### 15.4 Method Detection Limit

The method detection limit (MDL) is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is not zero. It is computed as follows from data obtained by repeatedly determining an analyte in a given sample matrix:

- 1. Analyze at least seven samples of a homogeneous matrix spike that contains the analyte(s) of interest at concentrations of three to five times the expected MDL. The entire sample preparation and analysis protocol must be applied in each analysis; simply preparing one sample and repeating a measurement three or more times on the sample in not acceptable.
- 2. Compute the standard deviation of the results for each analyte.
- 3. Compute the MDL using the following equation:

$$MDL = t_{(n-1, \alpha=0.99)}(s)$$

Where t is the one-sided student's t value appropriate for the number of samples analyzed, n;  $\alpha$  is the statistical confidence level; and s is the standard deviation.

The one-sided *t*-values are presented below:

Number of samples	<i>t</i> -value	
7	3.14	
8	3.00	
9	2.90	
10	2.82	

# 15.5 Linear Dynamic Range:

The linear dynamic range is the concentration range over which the instrument response is linear. It is determined by analyzing a series of standard solutions that extends beyond the non-linear calibration region at both the low and high extremes, and selecting that range of standards which demonstrates a linear relationship between instrument response and concentration.

### 16.0 CORRECTIVE ACTION

An essential element of the QA Program, Corrective Action provides systematic, active measures taken in the resolution of problems and the restoration of analytical systems to their proper functioning.

Corrective actions for laboratory problems are described in MITKEM Corporation laboratory operating manuals. Personal experience often is most valuable in alerting the bench scientist to questionable results or the malfunctioning of equipment. Specific QC procedures are designed to help the analyst determine the need for corrective actions (see Section 11, Data Reduction, Validation and Reporting). Corrective actions taken by scientists in the laboratory help avoid the collection of poor quality data.

Examples of conditions that warrant corrective actions are:

- 1. Tuning or calibrations of instruments fall outside of specifications.
- 2. QC data for precision and accuracy lie outside of acceptance limits.
- 3. Undesirable trends develop in concentration, surrogate and spike recoveries, response factors or relative percent difference.
- 4. Abnormal variation in detection limits.
- 5. Check sample results out of range.

Problems not immediately detected during the course of analysis may require more formalized, long-term corrective action. The essential steps in MITKEM Corporation corrective action system are:

- 1. Identify and define the problem.
- 2. Assign responsibility for investigating the problem.
- 3. Investigate and determine the cause of the problem.
- 4. Determine a corrective action to eliminate the problem.
- 5. Assign and accept responsibility for implementing the corrective action.
- 6. Establish effectiveness of the corrective action and implement it.
- 7. Verify that the corrective action has eliminated the problem.
- 8. Document the actions taken and those planned.

This scheme is generally accomplished through the use of Corrective Action Request Forms (Figure 16-1) available to all MITKEM staff members. Using this form, any laboratory scientist or project member may notify the QA Director of a problem as described in SOP No. Q07. The QA Director initiates the corrective action by relating the problem to the appropriate laboratory managers and/or project managers who then investigate or assign responsibility for investigating the problem and determine its cause. Once determined, an appropriate corrective action will be approved by the QA Director. Its implementation is later verified through and internal laboratory audit.

Information contained on corrective action forms is kept confidential within MITKEM and is generally limited to the individuals involved. Severe problems and difficulties may warrant special reports to the President of MITKEM who will ensure that the appropriate corrective actions are taken.

## Nonconformance:

Any breech of standard protocols is a nonconformance item that is documented on the Corrective Action Request Form and management informed immediately. The following are nonconformance items:

- 1. Sample holding time exceeded.
- 2. Hoods, Class "S" weights, NIST Thermometers, balances, automatic pipetters, being used but not certified.
- 3. Expired standards being used.
- 4. Manual integration being misrepresented.

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Figure 16-1
Quality Assurance Corrective Action Request Form

# Figure 16-1 Quality Assurance Corrective Action Request Form

# MITKEM CORPORATION

# Quality Assurance Corrective Action Request

Originator:		Date:
Laboratory:		Project:
Problem:		·
Action Planned:		Date Implemented:
		•
Resolution:		•
QA/QC Director:		
QAT00001	CAR#	

# 17.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The MITKEM Quality Assurance Director submits a QA report each quarter to the President of MITKEM and the Technical Director of the Laboratory Division. The report is to be completed and submitted no later than the third week of the end of the quarter. The quarter months are March, June, September, and December. The report contains detailed information and QA activities during the previous three months including:

- 1. Summary of systems and performance audits.
- 2. Performance evaluation samples analyzed, and scores received.
- 3. Status of certifications.
- 4. Laboratory QA/QC reviews.
- 5. Problems and corrective actions.
- 6. Comments and recommendations.

In case of a severe problem or difficulty, a special report is prepared by the QA Director and submitted immediately to management.

# 18.0 SAFETY

MITKEM maintains safety program managed by the Health and Safety Officer, the Safety Committee Chairperson, and the Safety Committee. Responsibilities include many aspects with comply with the Right-to-Know Laws. Training includes:

- Training seminar with information on basic safety instruction, location of safety equipment, etc.,
- Chemical Hygiene Plan/Health and Safety manual,
- Centralized MSDS information,
- Maps with safety equipment and all exits noted, and
- Posted safety rules.

# MITKEM CORPORATION INSTRUMENTATION and EQUIPMENT LIST Updated 07/31/00

Instrumentation	Vendor and Model Number	Age (years)
2- GC System for Volatile Organics	Hewlett Packard Model 5890 GC with Tandem OI PID/HECD or PID/FID Detectors; with Tekmar Purge and Trap sample concentrators and Autosampler	2 and 5
3- GC/ECD Systems for Pesticides/ PCB's and Herbicides	Hewlett Packard Model 5890 GC with dual Electron Capture Detectors and Autosampler	2 to 5
1-GC/ECD System for Pesticides/ PCB's	Hewlett-Packard Model 6890 GC with dual Electron Capture Detectors and Autosampler	new
1-GC system for Petroleum Hydrocarbon Analysis and Fingerprinting	Hewlett Packard Model 6890 GC with dual towers, dual FID detectors and Autosampler	3
2- GC/MS Systems for Volatile Organics	Hewlett Packard Model 5890/5972 GC/MS with Tekmar Purge and Trap sample concentrator and autosampler /O-I Analytical 456 Concentrator and 4552 Autosampler	5
1- GC/MS System for Volatile Organic Analyses.	Hewlett-Packard Model 6890/5972 GC/MS System with OI 4560/DPM16 Purge and trap and Autosampler.	2
1-GC/MS System for Volatile Organic Analyses.	Hewlett-Packard Model 6890/5973 GC/MS System with OI 456/4552 Concentrator and Autosampler	new
2- GC/MS System for Semivolatile Organics Analysis	Hewlett-Packard Model 5890/5872 GC/MS with autosampler	2 and 5
1-GC/MS System for	Hewlett-Packard Model 6890/5973	new

Semivolatile Organics Analysis	GC/MS with autosampler	
High Performance Liquid Chromatography (HPLC) System	Perkin Elmer System with Variable UV Detector	3
Inductively Coupled Argon Plasma (ICAP) Spectrophotometer for Metals Analysis	Perkin Elmer Optima 3000XL Transaxial ICAP with autosampler	3
Inductively Coupled Argon Plasma (ICAP) Spectrophotometer for Metals Analysis	Perkin Elmer Optima 3100XL Transaxial ICAP with autosampler	1
Graphite Furnace Atomic Absorption Spectrophotometer for Low Level Metals Analysis	Perkin Elmer 4100ZL ZEEMAN with (GFAA) autosampler	5
Flow Injection Atomic Absorption Spectrophotometer (FIAS) for Mercury Analysis	Perkin Elmer FIAS with autosampler	5
Ion Autoanalyzer for automated wet chemistry analyses	LACHAT Quick Chem 8000 dual channel ion analyzer	2
2- Gel Permeation Chromatograph for Sample Cleanup for Organic Analyses	ABC/O. I. Analytical	New
Total organic carbon, total carbon, total inorganic carbon analyzer with water and soil analysis modules.	O.I. Corporation Model 1020 combustion TOC system.	2
Diskette and Forms Deliverable for CLP Organics	Target Software by ThruPut Corporation for Windows NT	3
Diskette and Forms Deliverable for CLP Inorganics	EDR System Software by Ward Scientific	3
50 Gigabyte Onstream tape drives	2ea	1

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Veritas Backup Exec	for Windows NT Multisystem	1
Analytical Balance	Denver Instrument Company Model 100A	3
Top-Loading Balance	Denver Instrument Company Model XE-510	1 - 4
	Model XP-3000 (X010176)	
	Model XP-3000 (X010122)	
	Ohaus, Model TS2KS	
	Ohaus, Model CT200	
	Ohaus, Model 5C6010	
	Ohaus, Model CT200	
Refrigerators/Freezers	R1, Walk-in	1 - 5
	R2, Kenmore	
	R/F3, Kenmore	
	R/F4, GE	
<b>\</b>	R/F5, Hotpoint	
	R/F7, Hotpoint	
	R/F8, GE	
	R/F9, GE	
	R/F10, Amana	
	R11, GE	
	R12, Diplomat	
	R/F13, Whirlpool	

	R14, Excellence	
Recirculator, refrigerated	Neslab, CFT-150	2
	Neslab, CFT-150, 3ea	1-6
Ovens	Fisher Scientific, Model 516G	1 – 6
	Precision, 25EG, 2ea	
	Fisher Scientific, Model 750F	
DI Water System	Barnstead, E-Pure	4
	Millipore	4
	US Filter	1
Hotplates and Stirrers	Fisher, Stirrer, 7ea	1 – 5
-	Fisher, Hotplate/Stirrer, 3ea	
	Thermolyne, Hotplate, 7ea	
Conductivity Meter	Hanna, Model HI 8733	1
PH Meters	Orion, Model 520A, 2ea With 4 Combination Electrodes	2
Water Baths	Precision	1
	Hot water baths, 2ea	2-5
Thermometers	NIST, Certified, 50 to 100°C	1
	NIST, Certified, 0 to 200°C, 2ea	2 – 4
	Oven, 0 to 2500C, 4ea	1 – 6
	Refrigerator, -5 to 150C, 13ea	1 – 5

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	Freezer, 0 to -300C, 9ea	1 – 5
Muffle Furnace	Paragon, Model DTC 800C	2
Spectrophotometers	Genesys, Spectronic 20	1
	Spectronic, Spectronic 20	6
Pepettors	Wheaton and Fisher, Adjustable	1 – 3
Centrifuge	10 – 100uL, 11ea IEC Centra, Model CL2	2
Computer laboratory information Management system to manage information from project quote through sample receipt, login, analysis, data collection, reporting and invoicing. Providesmanagement information, status tracking, QC charting and documentation.	ChemWare, Inc. Horizon Laboratory Information Management (LIMS) system for Windows.	2

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# MITKEM CORPORATION

# CONFIDENTIALITY, ETHICS AND DATA INTEGRITY AGREEMENT

I.		rity required of me with regard to the	ame), state that I understand the standards of he duties I perform and the data I report in	
	conne	ection with my employment at Mith	kem Corporation.	
II.	I agre	ee that in the performance of my du	ities at Mitkem Corporation:	
	A.	I shall not intentionally report da measured or observed;	ata values or results that are not the actual valu	es
	B.	I shall not modify data values ur through a measurable analytical	nless the modification can be technically justif process.	fied
	C.	I shall not intentionally report the true and actual dates and times o	e dates and times of data analyses that are not of analyses; and	the
~	D.	I shall not intentionally represent	t another individual's work as my own.	
III.	I agree to report immediately any accidental or intentional reporting of non-authentic d by myself. Such report must be made to any member of Mitkem Corporation's Management (Kin Chiu, Reinier Courant, James Bennett, Edward Lawler and Leonard Ranalli) both orally and in writing.			
IV.	I agree to report immediately any accidental or intentional reporting of non-authentic by other employees. Such report must be made to any member of Mitkem Corporatio Management (Kin Chiu, Reinier Courant, James Bennett, Edward Lawler, Karen Gav and Leonard Ranalli) both orally and in writing.			n's
V.	I agree not to divulge any pertinent information including but not excluded to data a information about a project to outside sources without the prior consent of the clien		i all	
		I that failure to comply with the abo diate dismissal from Mitkem Corpo	ove ethics and data integrity agreement can resoration.	ult
(Signa	iture)		(Date)	