Powell~Harpstead, Inc.

Professional Engineering Services

ADDITIONAL STANDARD OPERATING PROCEDURES (SOPs)

PROPERTY:

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MOUNT LAUREL, NEW JERSEY 08054

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*personal*DataRAM
models *p*DR-1000AN and *p*DR-1200

INSTRUCTION MANUAL

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CONTENTS

ONE YEAR LIMITED WARRANTY

THERMO ANDERSEN warrants to the original Purchaser that the apparatus to be delivered hereunder will be of the kind designated or specified and free of defects in workmanship or material (excluding rechargeable batteries and rechargeable battery packs). THERMO ANDERSEN makes no other express warranty, and disclaims any implied warranty of merchantability or fitness for purpose.

If the apparatus fails to conform to the above warranty, and notice is received by THERMO ANDERSEN from Purchaser within one year from the date of shipment, THERMO ANDERSEN will, at its option, either repair the defective part or parts or make available a repaired or replacement part. This warranty extends to all parts and labor involved in the required repair to the extent that said repair was not caused by negligence in operation of the apparatus by the Purchaser. THERMO ANDERSEN will perform the repair at its plant with all shipping and insurance costs paid by the Purchaser or, upon mutual consent of the parties, at a site designated by the Purchaser except, in the latter circumstances, the Purchaser will be responsible to reimburse THERMO ANDERSEN for all costs associated with travel, per diem and travel time of those THERMO ANDERSEN individual(s) deemed appropriate to effectuate the repair.

Repair or replacement of the apparatus in the manner and for the time period specified above, is the Purchaser's exclusive remedy and will satisfy all liabilities of THERMO ANDERSEN to Purchaser arising out of the supply or use of the apparatus, whether based on contract, warranty, negligence or otherwise. In no event will THERMO ANDERSEN be liable for any incidental or consequential loss or damage resulting from any failure of the apparatus to conform to the contract of sale.

Rechargeable batteries and rechargeable battery packs shall be warranted for 30 days to be free of defects in workmanship or material. During this 30 days rechargeable batteries and rechargeable battery packs that fail shall be returned to THERMO ANDERSEN for evaluation before warranty replacements are sent.

1.0 GENERAL DESCRIPTION

The MIE *personal*DataRAM™ (for Personal Data-logging Real-time Aerosol Monitor) is a technologically advanced instrument designed to measure the concentration of airborne particulate matter (liquid or solid), providing direct and continuous readout as well as electronic recording of the information.

The *personal*DataRAM is available in two versions: model *p*DR-1000AN and model *p*DR-1200. The model *p*DR-1000AN operates as a passive air sampler whereas the model *p*DR-1200 uses active air sampling. The user can convert from one to the other of these two versions by means of optional conversion kits offered by MIE, Inc. (see Sections 4.2 and 15.0 of this manual).

The model *p*DR-1000AN samples passively (i.e., without a pump) the air surrounding the monitor; air accesses freely the sensing chamber of the instrument by means of convection, diffusion, and adventitious air motion. The model *p*DR-1200, on the other hand, requires a separate air driver (not included) such as a personal-type pump for its operation.

In addition, the model *p*DR-1200 includes a particle size-selective inlet cyclone which permits size segregated measurements (i.e., PM_{10}, $PM_{2.5}$, respirable, etc.) as well as enables the user to perform aerodynamic particle sizing by varying the sampling flow rate. The model *p*DR-1200 incorporates, downstream of its photometric sensing stage, a standard 37-mm filter holder on which all sampled particles are collected for subsequent analysis or gravimetric referencing/calibration, if so desired.

The *personal*DataRAM is the result of many years of field experience acquired with thousands of units of its well known predecessor, the MIE MINIRAM, and embodies many technological advances made possible by the latest electronic hardware and software. The *personal*DataRAM is also a worthy miniaturized companion to the MIE DataRAM, a recognized paragon of portable aerosol monitors.

The *personal*DataRAM is a high sensitivity nephelometric (i.e. photometric) monitor whose light scattering sensing configuration has been optimized for the measurement of the respirable fraction of airborne dust, smoke, fumes and mists in industrial and other indoor environments.

The *personal*DataRAM is an ultra-compact, rugged and totally self-contained instrument designed for hand-held, belt-worn, as well as unattended operation. It is powered either by its internal replaceable battery, or by an optional attachable rechargeable battery pack, or by an AC supply (included as standard accessory). For the model *p*DR-1200, power to an adjunct pump must be provided separately.

Zeroing is accomplished by means of a hand-inflatable "zero air" pouch included with the model *p*DR-1000AN, and by an inlet filter cartridge provided with the model *p*DR-1200. In addition, the instrument automatically checks agreement with its original factory calibration by checking its optical background during the zeroing sequence.

The *personal*DataRAM covers a wide measurement range: from 0.001 mg/m^3 (1 µg/m^3) to 400 mg/m^3, a 400,000-fold span, corresponding to very clean air up to extremely high particle levels.

In addition to the auto-ranging real-time concentration readout, the *personal*DataRAM offers the user a wide range of information by scrolling its two-line LCD screen, such as run start time and date, time averaged concentration, elapsed run time, maximum and STEL values with times of occurrence, etc.

Operating parameters selected and diagnostic information displays are also available. Furthermore, the *personal*DataRAM features complete, large capacity internal data logging capabilities with retrieval through an externally connected computer. The stored information (up to 13,000 data points) includes average concentration values, maximum and STEL values with time information as well as tag numbers.

Selectable alarm levels with built-in audible signal and switched output, a RS-232 communications port, and a programmable analog concentration output (voltage and current) are all part of this versatile instrument.

A custom software package is provided with the *personal*DataRAM to program operating/logging parameters (e.g. logging period, alarm level, concentration display averaging time, etc.) as well as to download stored or real-time data to a PC or laptop for tabular and/or graphic presentation. If required, the data can also be imported to standard spreadsheet packages (e.g. Microsoft Excel™, Lotus 1-2-3™, etc.).

2.0 SPECIFICATIONS

- Concentration measurement range (auto-ranging)[1]: 0.001 to 400 mg/m^3
- Scattering coefficient range: 1.5 x 10^{-6} to 0.6 m^{-1} (approx.) @ λ =880 nm
- Precision/repeatability over 30 days (2-sigma)[2]:
  ± 2% of reading or ±0.005 mg/m^3, whichever is larger, for 1-sec. averaging time
  ±0.5% of reading or ±0.0015 mg/m^3, whichever is larger, for 10-sec. averaging time
  ±0.2% of reading or ±0.0005 mg/m^3, whichever is larger, for 60-sec. averaging time
- Accuracy[1]: ±5% of reading ±precision
- Resolution: 0.1% of reading or 0.001 mg/m^3, whichever is larger
- Particle size range of maximum response: 0.1 to 10 μm
- Flow rate range (model *p*DR-1200 only): 1 to 10 liters/minute (external pump required)
- Aerodynamic particle sizing range (model*p*DR-1200 only): 1.0 to 10 μm
- Concentration display updating interval: 1 second
- Concentration display averaging time[3]: 1 to 60 seconds
- Alarm level adjustment range[3]: selectable over entire measurement range
- Alarm averaging time[3]: real-time (1 to 60 seconds), or STEL (15 minutes)
- Datalogging averaging periods[3]: 1 second to 4 hours
- Total number of data points that can be logged in memory: 13,391
- Number of data tags (data sets): 99 (maximum)
- Logged data:
  - Each data point: average concentration, time/date, and data point number
  - Run summary: overall average and maximum concentrations, time/date of maximum, total number of logged points, start

time/date, total elapsed time (run duration), STEL concentration and time/date of occurrence, averaging (logging) period, calibration factor, and tag number.

- Elapsed time range: 0 to 100 hours (resets to 0 after 100 hours)
- Time keeping and data retention: › 10 years
- Readout display: LCD 16 characters (4 mm height) x 2 lines
- Serial interface: RS-232, 4,800 baud
- Computer requirements: IBM-PC compatible, 486 or higher, Windows™ '95 or higher, ≥ 8 MB memory, hard disk drive, 3.5" floppy, VGA or higher resolution monitor
- Outputs:
  * Real-time digital signal (1 sec^{-1}): concentration, 16-character code
  * Real-time analog signal: 0 to 5 V and 4 to 20 mA. Selectable full scale ranges of[3]: 0 - 0.1, 0 - 0.4, 0 - 1.0, 0 - 4.0, 0 - 10, 0 - 40, 0 - 100, and 0 - 400 mg/m^3.
    Minimum load impedance for voltage output: 200 kΩ.
    Maximum load impedance for current output: 300 Ω (when powered by AC power supply)
  * Alarm output: 1 Hz square wave, 5 V peak-to-peak amplitude. Load impedance > 100 kΩ
- Internal battery: 9V alkaline, 20-hour run time (typ.)
- Current consumption: 15 to 25 mA (in Run Mode); 10 to 20 mA (in Ready Mode)
- AC source: universal voltage adapter (included) 100-250 V~, 50-60 Hz (CE marked)
- Optional battery pack: model *p*DR-BP, rechargeable NiMH, 72-hour run time (typ.)
- Operating environment: -10° to 50° C (14° to 122° F), 10 to 95% RH, non condensing
- Storage environment: -20° to 70° C (-4° to 158° F)
- Dimensions (max. external):
  * Model *p*DR-1000AN: 153 mm (6.0 in) H x 92 mm (3.6 in) W x 63 mm (2.5 in) D

    * Model *p*DR-1200 (including cyclone and filter holder): 160 mm (6.3 in) H x 205 mm (8.1in) W x 60 mm (2.4 in) D

- Weight:
    * Model *p*DR-1000AN: 0.5 kg (18 oz)
    * Model *p*DR-1200: 0.68 kg (24 oz)

- Cyclone (included in model *p*DR-1200 only): BGI model GK 2.05

- Filter holder (included in model *p*DR-1200 only): Millipore type MAWP 037 AO (with 0.8 μm pore size filter)

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[1]Referred to gravimetric calibration with SAE Fine (ISO Fine) test dust (mmd = 2 to 3 μm, σ_g = 2.5, as aerosolized)

[2]At constant temperature and full battery voltage

[3]User selectable

3.0 USER GUIDELINES

3.1 Handling Instructions

The *personal*DataRAM is a sophisticated optical/electronic instrument and should be handled accordingly. Although the *personal*DataRAM is very rugged, it should not be subjected to excessive shock, vibration, temperature or humidity. As a practical guideline, the *personal*DataRAM should be handled with the same care as a portable CD player.

If the *personal*DataRAM has been exposed to low temperatures (e.g. in the trunk of a car during winter) for more than a few minutes, care should be taken to allow the instrument to return near room temperature before operating it indoors. This is advisable because water vapor may condense on the interior surfaces of the *personal*DataRAM causing temporary malfunction or erroneous readings. Once the instrument warms up to near room temperature, such condensation will have evaporated. If the *personal*DataRAM becomes wet (e.g. due to exposure to water sprays, rain, etc.), allow the unit to dry thoroughly before operating.

Whenever the *personal*DataRAM is shipped care should be taken in placing it in its carrying case and repackaging it with the original cardboard box with the factory provided padding.

3.2 Safety Instructions

- Read and understand all instructions in this manual.
- Do not attempt to disassemble the instrument. If maintenance is required, return unit to the factory for qualified service.
- The *personal*DataRAM should be operated only from the type of power sources described in this manual.
- When replacing the internal 9-V battery, follow the instructions provided on the back panel of the unit.
- Shut off *personal*DataRAM and any external devices (e.g. PC) before connecting or disconnecting them.
- Shut off *personal*DataRAM before replacing the internal battery, or when plugging in or disconnecting the AC power supply or the optional rechargeable battery pack.

3.3 Handling and Operation

3.3.1 Model *p*DR-1000AN

The model *p*DR-1000AN can be operated in any position or orientation. Exposure to high intensity fluctuating light of the interior of the sensing chamber, through the

front and back slotted air openings (see Section 5.5), should be avoided. Such large intensity transients may cause erroneous readings. Direct access of sunlight to the sensing chamber should be prevented.

Typical modes of instrument support/handling include:

- Hand-held. Do not obstruct or cover the sensing chamber opening slots on front and back of unit.
- Belt attached. Use belt clip provided as standard accessory. The unit can be worn on a waist belt, or with optional shoulder belt (model *p*DR-SS) for breathing zone monitoring.
- Table top operation. The *p*DR-1000AN can be placed on a table either in an upright position (i.e., resting on its lower protective bumper), or on its back (i.e., resting on the rear edges of its two protective bumpers).
- Tripod mounted. The unit can be attached to any standard tripod using the threaded bushing on the bottom of the monitor (see Figure 3).
- Fixed point operation. The model *p*DR-1000AN can be mounted at a fixed location (e.g., wall or post) using the optional wall mounting bracket, model *p*DR-WB.

3.3.2 Model *p*DR-1200

The *p*DR-1200 requires an external air suction device, such as a small diaphragm pump (e.g., model *p*DR-PU) for its sampling operation. The inlet of the pump must be connected by means of tubing to the hose fitting on the *p*DR-1200 filter holder attached to sensing chamber (see Figure 2).

The inlet metal tube of the cyclone can be oriented in any desired direction (i.e., upward, forward, downward or backward) by rotating the cyclone body within its holder cup on the right side of the sensing chamber (see Figure 2).

Always ensure unobstructed access to the cyclone inlet when sampling directly the air in the instrument's vicinity. Alternatively, tubing can be connected to the cyclone inlet in order to extract a sample stream from a duct, chamber or other enclosed volume.

Typical modes of instrument support/handling include:

- Hand-held. For example, using a personal type pump, clipped to the belt and using a tubing connection to the *p*DR-1200.
- Belt attached. Use belt clip kit provided as standard accessory. The unit can be worn on a waist belt, or with the optional shoulder belt (model *p*DR-SS) for breathing zone monitoring. A personal pump can then be belt-worn as well.
- Table top operation. The *p*DR-1200 can be placed on a table either in an upright position (i.e. resting on its lower protective bumper), or on its back (i.e. resting on its backside).
- Tripod mounted. The unit can be attached to any standard tripod using the

threaded opening on the bottom base (see accessory attachment fitting on Fig. 4).

- Wall mounted for fixed point monitoring. Use optional wall mounting bracket, model *p*DR-WB, either in combination with model *p*DR-PU pump module and model *p*DR-AC power supply (powering both the *p*DR-1200 and the *p*DR-PU), or with a separate pump.

3.4 Air Sampling Guidelines

Although the *personal*DataRAM is designed primarily for intramural use, i.e. for indoor air quality, in-plant, or mining environment monitoring, its active sampling version (model *p*DR-1200) also makes it compatible with extramural use (i.e. ambient monitoring). General ambient monitoring applications, however, are performed preferentially using an appropriate inlet configuration, in order to ensure representative particle sampling under conditions of variable wind speed and direction. Consult with MIE for such outdoor applications.

For typical area monitoring applications, the *personal*DataRAM should be placed and operated centrally within the area to be monitored, away from localized air currents due to fans, blowers, ventilation intakes/exhausts, etc. This is to ensure representative sampling within the area to be assessed.

3.5 Environmental Constraints and Certifications

The *personal*DataRAM is designed to be reasonably dust and splash resistant, however, it is not weatherproof. To operate the unit outdoors provisions should be made to protect it from environmental extremes outside its specified range, and from any exposure to precipitation.

The *personal*DataRAM is certified for compliance with the electromagnetic radiation limits for a Class A digital device, pursuant to part 15 of the FCC Rules. The unit also complies and is marked with the CE (European Community) approval for both immunity to electromagnetic radiation and absence of excessive emission interference.

4.0 ACCESSORIES

4.1 Standard Accessories

The *personal*DataRAM is provided to the user with the following standard accessories:

- Soft-shell carrying case (MIE model *p*DR-CC-1)
- Digital communications cable (MIE model *p*DR-DCC)
- Analog signal/alarm output cable (MIE model *p*DR-ANC)
- Communications software disk (MIE model *p*DR-COM)
- Z-Pouch zeroing kit (MIE model *p*DR-ZP)(for use with *p*DR-1000AN only)

- Zeroing filter cartridge and tubing (MIE model *p*DR-ZF)(for use with *p*DR-1200 only)
- Belt clip kit (MIE model *p*DR-CA)
- AC power supply (and charger for optional MIE model *p*DR-BP) (MIE model *p*DR-AC)
- Metal cyclone (MIE model *p*DR-GK2.05)(for use with *p*DR-1200 only)
- 37-mm filter holder and hose fitting (MIE model *p*DR-FH)(for use with *p*DR-1200 only)
- Instruction manual

4.2 Optional Accessories

The following optional accessories are available from MIE for use with the *personal*DataRAM:

- Rechargeable battery module (MIE model *p*DR-BP)
- Shoulder strap (MIE model *p*DR-SS)
- Remote alarm unit (MIE model *p*DR-RA)
- Wall mounting bracket (MIE model *p*DR-WB)
- Active sampling kit to convert model *p*DR-1000AN to model *p*DR-1200 (MIE model *p*DR-ASC)
- Upper bumper kit to convert model *p*DR-1200 to model *p*DR-1000AN (MIE model *p*DR-UB)
- Attachable pump unit (MIE model *p*DR-PU)(for use with *p*DR-1200 only)

5.0 INSTRUMENT LAYOUT

The user should become familiar with the location and function of all externally accessible controls, connectors and other features of the *personal*DataRAM. Refer to Figures 1 through 6.

All user related functions are externally accessible. All repair and maintenance should be performed by qualified MIE personnel. Please contact the factory if any problem should arise. Do not attempt to disassemble the *personal*DataRAM, except as described in Section 12.0 (Maintenance), **otherwise voiding of instrument warranty will result.**

5.1 Front Panel

Refer to Figures 1 (for model *p*DR-1000AN) or 2 (for model *p*DR-1200) for location of controls and display.

The front panel contains the four touch switches (keys) and the LCD screen required for the operation of the *personal*DataRAM.

The four touch switches provide tactile ("popping") feedback when properly actuated.

The ON/OFF key serves only to turn on the unit (while it is in the off state), and to turn it off (when it is operating).

The EXIT and ENTER keys serve to execute specific commands that may be indicated on the screen, and the NEXT key generally serves to scroll the displayed information, e.g. to review the operating parameters that have been programmed, display maximum/STEL values, diagnostic values, etc.

If an incorrect command is keyed (e.g. ENTER when the *personal*DataRAM displays real-time concentration) a beep is heard to alert the user.

The two-line, 16-character per line LCD indicates either measured values of concentration (instantaneous and time averaged on the same screen), elapsed run time, maximum and STEL (short term excursion limit) values, operating and logging parameters, diagnostics, or other messages.

The acoustic alarm transducer is located directly behind the center of the MIE arrow logo on the front panel.

5.2 Bottom Base

Refer to Figures 3 (for model *p*DR-1000AN) or 4 (for model *p*DR-1200). The base of the *personal*DataRAM contains the following: a) internal battery compartment cover, b) external DC power input receptacle, and c) threaded bushing for the attachment of optional battery pack, tripod, or other mounting/support hardware.

Only the internal battery compartment cover should be opened by the user, for removal and replacement of the on-board 9-V battery. Removal of the base plate could result in voiding of instrument warranty.

5.3 Right Side Panel

Refer to Figures 5 (for model *p*DR-1000AN) or 6 (for model *p*DR-1200) which shows the manner of attachment of the belt clip assembly (belt clip should be attached only if required by the user). The right side panel (as viewed from front panel) contains the 6-contact modular jack connector receptacle for digital (RS-232) communications and analog signal output. This connector also provides the alarm output control for a remote/auxiliary alarm signal. The contacts (from top to bottom) are:

1: 4 - 20 mA analog output (positive)
2: Alarm output
3: Digital data transmission
4: Digital input

5: Common ground (signal returns)
6: 0 to 5 V analog output (positive)

The digital communications cable provided as a standard accessory is to be inserted into this receptacle for interconnection to a computer (for data downloading or to reprogram parameters). The analog output cable is provided with flying leads for interconnection with other data processing and/or control systems.

WARNING: The modular jack receptacle on the side of the *personal*DataRAM should be used only for communications with computers and alarm circuitry. **Do not, under any circumstance, connect any communications equipment (e.g., telephone) to this receptacle.**

5.4 Back Panel and Belt Clip

The back panel consists of a label with important user information on safety procedures and certifications, model and serial numbers, etc.

The back panel is provided with mounting hardware for the attachment of the belt clip kit (see Figures 5 or 6 for mounting configuration of the belt clip).

5.5 Sensing Chamber

Referring to Figure 1 or 2, the upper mid-section of the *personal*DataRAM contains the optical sensing chamber. This chamber is the only internal section that the user should access for maintenance purposes (see Section 12.2).

On the model *p*DR-1000AN, air enters the sensing chamber through the two slot shaped inlets (one on the front and other on the back) under the protective bumper. During instrument operation those two openings should remain unobstructed in order to ensure free access of the surrounding air. When the model *p*DR-1000AN is used as personal monitor, i.e., clipped to a person's belt, the rear air inlet opening may be partially obstructed, but care should be exercised in ensuring that the front air inlet remains free of any obstructions.

On the model *p*DR-1200, air enters the sensing chamber through the opening in the cyclone receptacle cup (black cup on right side of sensing chamber), passes through the photometric stage, and exits through the opening in the filter holder receptacle cup (black cup on left side of sensing chamber), after which the air passes through the filter.

6.0 PREPARATION FOR OPERATION

6.1 Battery Installation

When shipped from the factory, the *personal*DataRAM will arrive without its replaceable 9V battery installed. Two fresh alkaline batteries are factory packed

separately in the carrying case, one of which should be installed in the *personal*DataRAM when preparing it for operation.

NOTE: Whenever the *personal*DataRAM is to be left unused for an extended time (i.e. longer than a month), the 9V battery should be removed from the unit.

Removing the battery will lose neither the program, time/date keeping, nor stored data.

To install the battery proceed as follows:
- Hold the *personal*DataRAM upside down.
- Loosen thumbscrew that secures the battery compartment cover (see Figure 3 or 4), and remove that cover.
- Observe battery polarity and the back panel battery orientation pattern (the negative battery terminal is the one closer to the side of the instrument).
- Insert the battery by sliding it in until it bottoms out. It should protrude slightly above the bottom surface of the instrument.
- Place battery compartment cover over battery and, while pushing down the cover firmly (taking care that the cover seats flush on the bottom surface of the *personal*DataRAM), tighten thumbscrew securely.

6.2 Battery Replacement

Normally, only alkaline type 9V batteries (type 1604A, or equivalent) should be used with the *personal*DataRAM.

Only fresh batteries should be used in order to ensure the maximum operating time. The *personal*DataRAM shuts itself off whenever the battery voltage falls below 6 volts (while retaining all programming and data). A fresh 9V alkaline battery, at room temperature, should provide typically 20 hours of continuous operation (please note that not all manufacturers produce batteries of equal capacity). Intermittent operation should extend the total running time because of partial battery recovery effects.

The approximate remaining battery capacity is indicated by the *personal*DataRAM (see Section 8.2) in increments of 1%, starting from 99%. If the remaining battery capacity is 40% or less, immediate restarting after shut off is automatically inhibited to prevent incomplete runs. If, nevertheless, a new run is to be initiated with low remaining battery capacity, do not shut off the *personal*DataRAM at the end of the previous run (i.e., remain in the Ready Mode, see section 7.0).

When significantly extended operating times are required (beyond the typical 20 hours), the use of either lithium or zinc-air batteries can be considered. The use of such alternative battery types can provide about 2 to 3 times longer operation than alkaline batteries.

6.3 AC Power Supply

A universal line voltage AC to DC power supply (MIE model *p*DR-AC) is provided as standard accessory with the *personal*DataRAM. This power supply can be used with any line with a voltage between 100 and 240 VAC (50 to 60 Hz). When using that power supply, its output plug should be inserted into the external DC receptacle at the base of the *personal*DataRAM (see Figure 3 or 4). Insertion of that connector automatically disables the internal 9V battery of the instrument. Removal of the *p*DR-AC plug from the instrument automatically re-connects the internal 9V battery.

NOTE: Before plugging in or unplugging the external power supply, the *personal*DataRAM must be shut off.

6.4 Rechargeable Battery Module

A rechargeable battery pack (MIE model *p*DR-BP) is available as an optional accessory. This unit attaches directly to the base of the *personal*DataRAM.

The *p*DR-BP contains a sealed nickel-metal-hydride battery, which provides typically 72 hours of continuous operation between successive charges (for 3-hour charging).

The use of the *personal*DataRAM, in combination with the *p*DR-BP connected to the a.c. power line ensures totally uninterruptible operation over indefinitely long time. In this operating mode, line power interruptions lasting up to 72 hours have no effect on measurement run continuity.

To attach the *p*DR-BP to the *personal*DataRAM, the instrument should be shut off. Carefully plug the *p*DR-BP into the external DC RECEPTACLE on the *personal*DataRAM. Rotate the large thumbscrew at the opposite end of the *p*DR-BP tightening it firmly. The *p*DR-BP can be recharged by means of the AC power supply of the *personal*DataRAM.

Detailed instructions for the use of the rechargeable battery module are furnished with that accessory.

6.5 Zeroing the *personal*DataRAM

One of the most important steps to be performed by the user before initiating a measurement run with the *personal*DataRAM is to zero the instrument. This is required to ensure maximum accuracy of concentration measurements, especially at low levels, i.e. below about 0.1 mg/m^3.

During the 2-minute pre-run automatic zeroing sequence (see Section 8.1), the *personal*DataRAM registers its own optical background, stores that level in its

memory, and then subtracts that background from all measured concentration values, until the zero is updated again by the user.

Although zeroing can be performed as often as desired (e.g., before every run), in practice it should not be necessary to do so more than once-a-month or even less frequently, except if average particulate concentrations should exceed about 0.5 mg/m^3.

6.5.1 Zeroing the model *p*DR-1000AN

Zeroing of the model *p*DR-1000AN requires a particle-free environment such as a clean room, clean bench, duct or area directly downstream of a HEPA filter, or the *p*DR-1000AN Z-Pouch (standard accessory). In some cases, a very clean, well air conditioned office may offer a sufficiently low particle concentration environment (i.e., $\leq 5\ \mu g/m^3$) for zeroing, as determined by another monitor (e.g., MIE DataRAM).

To zero the model *p*DR-1000AN by means of its Z-Pouch, proceed as follows:

- Wipe the outside surfaces of the *p*DR-1000AN to remove as much dust from those surfaces as possible before placing the instrument inside the Z-Pouch.
- In a reasonably clean environment, open the zipper of the Z-Pouch and place the *p*DR-1000AN inside it. Close the zipper shut.
- Open the small nipple on the Z-Pouch, and insert the fitting of the hand pump/in-line filter unit into the nipple.
- Start pumping the hand-pump until the Z-Pouch begins to bulge, and proceed with the steps in Section 8.1, pressing the keys of the instrument through the wall of the Z-Pouch. Then continue pumping.
- After completing the zeroing (step 2. of Section 8.1) procedure, open the Z-Pouch zipper and remove the *p*DR-1000AN. Close the zipper and flatten the Z-Pouch while plugging its nipple, in order to prevent dust contamination of the interior of the Z-Pouch.
- The *p*DR-1000AN is now zeroed and ready for a measurement run.

6.5.2 Zeroing the model *p*DR-1200

To provide the particle-free air required to zero the *p*DR-1200, either of two methods can be used: a) place the instrument on a clean-air bench or in a clean room, or b) connect to the cyclone inlet the green zeroing filter cartridge supplied with the *p*DR-1200. In either case, proceed as follows:

- After implementing either of the two methods, above, run the attached pump for at least one minute (e.g., at 4 liters/minute), and then proceed as described in Section 8.1 of this instruction manual, while continuing to run the pump (or leaving the unit in the clean air environment).

- Once the CALIBRATION: OK message appears on the *p*DR-1200 display, stop the pump and disconnect the zeroing filter cartridge from the cyclone inlet (or remove *p*DR-1200 from clean bench/room).
- The *p*DR-1200 is now zeroed and ready for a measurement run.

Note: While the *p*DR-1200 is used to monitor high dust concentrations (≥ 0.5 mg/m³), the flow through its sensing chamber should not be stopped before purging it, which can be done by connecting the green zeroing filter to the cyclone inlet and continuing to run the pump for about 2 minutes before shutting it off. This is to prevent dust contamination of the sensing chamber.

6.6 *p*DR-1200 Filter Holder Installation

The 37-mm filter holder provided with the *p*DR-1200 must be installed before operation of the instrument, in order to connect a sampling pump. To install the filter holder, remove protective cover, and insert the open collar over the black attachment cup with the external o-ring, on the left side of the *p*DR-1200 sensing chamber. Ensure complete insertion.

To replace the membrane filter separate the two sections of the plastic holder prying them apart with screwdriver or a coin. Make sure to place backing under the membrane filter before rejoining the two plastic rings.

7.0 OPERATING MODES

The *personal*DataRAM has several different operating modes which will be described in what follows. The specific commands and displays within each of these operating modes will be explained in detail in Section 8.0. A complete flow chart of keystrokes and screens is provided in Section 16.0.

7.1 Start-Up Mode

The *personal*DataRAM enters the Start-Up Mode as soon as the instrument is switched on. The user then has the choice to:
a) Wait before proceeding;
b) Zero the instrument and check its readiness; or
c) Proceed directly to the Ready Mode.

7.2 Ready Mode

Once the *personal*DataRAM is in the Ready Mode, the user is presented with the following alternatives:
a) Start a run immediately, or after any of the subsequent steps;
b) Review (by scrolling the display) all operating parameters, status and diagnostic data;

c) Activate or deactivate the logging function; activate, select (instantaneous or STEL), or deactivate alarm;
d) Program parameters or output logged data through a computer.

7.3 Run and Logging Mode

The Run Mode is the measurement/logging mode. The user can operate the *personal*DataRAM in this mode either with or without data logging. For example, the instrument may be used first as a survey monitor without logging, for walk-through assessment of an industrial plant, before deciding where to set up the unit for continuous monitoring and logging.

7.3.1 Data Logging

In order to activate the logging function, the unit must be in (or returned to) the Ready Mode (see Section 8.2).

If data logging has been enabled, the data will be logged in the next free (unrecorded) tag or data set. For example, if data had been recorded previously in tags # 1, 2 and 3 then, when a new run is initiated, the new data will be stored in tag #4. The data can be separated into number of sets (tags) up to a total of 99.

Any number of individual data points can be stored in a given tag, i.e. up to a maximum of 13,000 points (i.e. the total memory capacity of the *personal*DataRAM) assuming that no other data had been logged in other tags. This means that the total memory capacity of 13,000 data points can be grouped into any number of the available 99 data sets (tags).

7.3.2 Clearing of Memory

Data recorded in the *personal*DataRAM memory can be erased either through an external PC command using the MIE *p*DR-COM Custom Communications software provided as a standard accessory, or resetting the instrument (see Section 8.5). The PC method permits to erase the data in any number of selected tags, whereas the resetting method results in the deletion of all data stored in the *personal*DataRAM.

7.3.3 Run Mode Display and Commands

When a measurement run has been initiated (see Section 8.3), the user has the following display choices:
a) Instantaneous and time-averaged concentrations (both on the same screen);
b) Elapsed run time, and run start time and date (both on the same screen);
c) Maximum displayed concentration from run start, and time/date at which current maximum occurred;
d) Short term excursion limit (STEL) from run start, and time/date at which current STEL occurred;
e) Remaining battery charge, and (if logging function is enabled) remaining free memory.
f) Analog output concentration range (if enabled)

The user can command the termination of the run at any time returning it to the Ready Mode. To download logged data into a PC, the *personal*DataRAM must be in the Ready Mode. No changes in the program parameters or operating conditions can be made while in the Run Mode.

The *personal*DataRAM can be shut off from any of the three operating modes. Even if shut off while in the Run Mode, the instrument will save all stored data.

8.0 OPERATION

8.1 Start-Up

	KEY	DISPLAY	NOTES
1.	**ON/OFF**	START ZERO:ENTER GO TO RUN: NEXT	Before starting a run with the *personal*DataRAM, zero it (see Section 6.5) and key **ENTER** while the unit is exposed to particle-free air. Alternatively, key **NEXT** to go to RUN/READY mode. If **ENTER** is keyed:
2.	**ENTER**	ZEROING V2.00	Keep clean air flowing while ZEROING is displayed* for 1.1 min., followed by one of these screens:
		CALIBRATION: OK	or,
		BACKGROUND HIGH	or,
		MALFUNCTION	If CALIBRATION: OK, then go to step 3. If one of the other two screens is displayed, consult Section 12.0.
3.	**NEXT**	START RUN: ENTER READY: NEXT	To start a measurement run key **ENTER** (Section 8.3, step 1). To set up for a run and scroll logging/ operating parameters, key **NEXT** (see Section 8.2).
4.	**ON/OFF**	TURN OFF PDR? Y:ENTER N:NEXT	Keying **ON/OFF** while the unit is operating will elicit this message to prevent accidental shut off. To confirm shut down, key **ENTER**. To continue operation, key **NEXT**.

*The number following the V on the screen refers to the installed firmware version.

8.2 Setting Up For A Run (Ready Mode)

	KEY	DISPLAY	NOTES
1.	**NEXT**	LOGGING DISABLED	This screen indicates the logging status. **To enable the logging function**, key **ENTER**. Toggling of the on/off logging status can be done by keying **ENTER**.
2.	**ENTER**	LOG INTRVL 600s TAG#: 4	This message indicates that logging is enabled. Example is for 10-min log period, selected through the PC (see Section 9.0), and next free tag is #4.
3.	**NEXT**	ALARM: OFF	This screen indicates the alarm status. Keying **ENTER** repeatedly toggles through the 3 alarm modes:
4.	**ENTER**	ALARM: INSTANT LEVEL:1.50 mg/m3	This enables the alarm based on the real-time concentration. The level (e.g. 1.50 mg/m3) must be set on the PC.
5.	**ENTER**	ALARM: STEL LEVEL:0.50 mg/m3	This enables the alarm based on the 15-min STEL value. The level (e.g. 0.50 mg/m3) must be set on the PC.
6.	**NEXT**	ANALOG OUTPUT: DISABLED	This screen indicates the analog signal output status. Keying **ENTER** will enable the analog output. Toggling the analog output on/off can be done by keying **ENTER**:
7.	**ENTER**	ANALOG OUTPUT: 0 - 0.400 mg/m3	This enables the analog output. The concentration range (e.g., 0 - 0.400 mg/m^3) must be set on the PC.
8.	**NEXT**	CAL FACTOR: 1.00 DIS AVG TIME 10s	This screen displays the calibration factor and the display averaging time. Both values can be edited via PC.

9.	**NEXT**	BATTERY LEFT 83% MEMORY LEFT 96%	This screen displays the remaining battery charge, and the remaining percentage of free memory.
10.	**NEXT**	CONNECT TO PC	When this screen has been selected, the operating parameters can be edited and/or the logged data can be downloaded via the PC (see Section 9.0). If **NEXT** is keyed again, the screen returns to RUN/READY:
11.	**NEXT**	START RUN: ENTER READY: NEXT	The instrument is now ready to run following the procedure in section 8.3.

8.3 Measurement Run Procedure

	KEY	DISPLAY	NOTES
1.	**ENTER**	LOGGING DISABLED	or, if logging was enabled:
		LOG INTRVL 600s TAG #: 4	Logging status will be displayed for 3 seconds.
		CONC*0.047 mg/m3 TWA 0.039 mg/m3	After a 3-second delay, the concentration screen appears (values shown here are examples). CONC is the real-time and TWA is the time-averaged concentration. **The * appears only if logging has been enabled.**
2.	**EXIT**	TERMINATE RUN? Y:ENTER N:EXIT	To terminate the current run and return to the Ready Mode, key **ENTER**. To continue the run, key **EXIT**.
3.	**EXIT**	CONC*0.047 mg/m3 TWA 0.039 mg/m3	Keying **NEXT** successively scrolls the display to show various run values (elapsed run time, maximum, STEL, etc.). Keying **EXIT** from any of those screens returns to the concentration display.

4.	**NEXT**	ET 06:12:49 ST 08:18:26MAY15	This screen shows the elapsed run time (ET) and the run start time/date (ST).
5.	**NEXT**	MAX: 0.113 mg/m3 T 10:08:44 MAY15	This screen shows the maximum concentration of current run and time/date of occurrence.
6.	**NEXT**	STEL:0.058 mg/m3 T 09:59:22 MAY15	This screen shows the 15-min STEL value of the current run and the time/date of occurrence.
7.	**NEXT**	BATTERY LEFT 83%	or, if logging was enabled:
		BATTERY LEFT 83% MEMORY LEFT 96%	This screen shows the amount of usable charge left in the battery and, if logging has been enabled, the overall amount of free memory left.
8.	**NEXT**	ANALOG OUTPUT: 0 - 0.400 mg/m3	This screen shows the status of the analog signal output, and the range, if this output has been enabled.
9.	**NEXT**	CONC*0.047 mg/m3 TWA 0.039 mg/m3	The last **NEXT** command returns the display to the concentration screen.
10.	**EXIT**	TERMINATE RUN? Y:ENTER N:NEXT	As indicated in step 2, to end current run, key **ENTER**, to return to the Ready Mode:
11.	**ENTER**	START RUN: ENTER READY: NEXT	This keystroke terminates the current run and returns the unit to the Ready Mode.

If during a run the instrument memory is filled completely, or if all 99 tags have been used, the run is automatically terminated and the display will indicate:

RUN TERMINATED
FULL MEMORY

If a new run is initiated after the memory has been filled, the *personal*DataRAM can be operated only as a monitor without logging. The memory must then be cleared (see Section 7.3.2) first before logging can be enabled again.

8.4 Abbreviated Run Start/Stop Instructions

To power-up and start a measurement run without zeroing and without logging, proceed as follows:

- Key sequentially **ON/OFF**, **NEXT** and **ENTER**.

To terminate run and shut down, proceed as follows starting from the concentration screen (otherwise key **EXIT** first):

- Key sequentially **EXIT**, **ENTER**, **ON/OFF** and **ENTER**.

8.5 Resetting Procedure

The *personal*DataRAM memory can be reset through commands entered on its own keypad (i.e. without requiring a PC).

Resetting accomplishes the following:

- Erases all stored data from memory;
- Resets all parameters and operating conditions to their default values and conditions; and
- Cancels the zero correction offset.

The procedure to reset the instrument is as follows:

Starting with the unit shut off, press the EXIT and ENTER keys at the same time, and while holding down those two keys, press ON. The screen will then indicate: PDR SELF-TEST... and several diagnostic screens will appear in rapid sequence (see Section 16.0, *Resetting/Electronics Checking Mode*), ending in the message TESTING COMPLETE. Shut off unit. When turned on again, the *personal*DataRAM memory will have been reset, as described above.

The **default** values and operating conditions of the *personal*DataRAM are:

- Logging period (LOG INTRVL): 60 seconds
- Logging status: disabled (LOGGING DISABLED)
- Alarm level: 1 mg/m^3
- Alarm status: disabled (ALARM: OFF)
- Analog output: 0 to 4 mg/m^3
- Analog output status: disabled (ANALOG OUTPUT :DISABLED)
- Real-time display averaging time (DIS AVG TIME): 10 seconds
- Calibration factor (CAL FACTOR): 1.00

When turning on the *personal*DataRAM after resetting the instrument, it should be zeroed (see steps 1. and 2. of Section 8.1) before a run is initiated. Otherwise, its internal optical background level will not be subtracted from the indicated concentration readings. Alternatively, if the instrument is not zeroed after resetting, it will indicate its unsubtracted optical background when run under particle free conditions.

9.0 COMMUNICATIONS WITH COMPUTER

9.1 Hardware and Software Requirements

The computer requirements to install the software provided with the *personal*DataRAM (MIE *p*DR-COM) are the following:

- IBM-PC compatible
- 486 or better processor
- Minimum operating system: Windows 95™ or better
- ≥ 8 MB of RAM
- 2 MB hard disk drive
- 3.5" floppy drive
- VGA or higher resolution monitor

NOTE: When large files are logged in the *personal*DataRAM in one single tag, a faster computer speed is required to handle the data. For example, if all 13,000 data points are logged in one tag, a Pentium I or II processor with a minimum speed of 166 MHz will be required. If, however, the maximum number of data points per tag is 1,000 or below, a 33 MHz, 486 DX processor will suffice.

MIE custom hardware and software (provided as standard accessories):

- Digital communications cable (MIE model *p*DR-DCC)
- Software floppy disk (3.5", MIE model *p*DR-COM)

9.2 Software Installation Procedure

To install the MIE provided software in the computer, proceed as follows:

1. Insert the 3.5" disk labeled "*p*DR-COM" into computer.

2. For Windows 95™ users, select **Start** and then **Run**. For Windows 3.1 and 3.11 users, from Program Manager select **File** and then **Run**.

3. Type in on the **Command Line: a: install** (or **b: install**, as required).

4. The message "**Do you wish to install *p*DR-COM?**" will appear. Click **OK** to continue, or **Cancel**.

5. A message appears allowing the option to change the default directory: "**C:\PDRCOM**". It is advisable to leave the default directory (unless you address the hard drive by a different letter), and select **OK**.

6. After a successful installation, the message "**Installation Complete!**" will appear.

9.3 Communication Between *personal*DataRAM and Computer

To effect the communication between the *personal*DataRAM (via the *p*DR-COM software installed in the computer as described in the preceding section) and the PC proceed as follows:

1. Connect the *personal*DataRAM to one of the computer's serial ports using the *p*DR-DCC cable provided by MIE. This cable has a 9-pin female connector for the computer port.

2. Key **ON** the *personal*DataRAM and then key **NEXT** repeatedly until CONNECT TO PC is displayed on the *personal*DataRAM.

3. On the computer, double click on the ***p*DR-COM** icon. A four-tabbed notebook display should appear. Click on the **Com Port Select** and select the port to which the *p*DR-DCC cable has been connected.

4. From the four-tabbed notebook displayed on the computer screen select the tab with the desired option. The options are:

- **Main**: This page allows the user to input the *personal*DataRAM serial number (or any other desired label), and select the Serial Com Port.

- **Logged data**: This page allows the user to download, tabulate, print data, or transfer to a CSV file the data stored in the *personal*DataRAM. This page also serves to display real-time numerical data when the computer is connected to the *personal*DataRAM in the Run Mode.

- **Graph data**: This page enables the downloading and graphing of stored data to the computer screen and to a printer. In the Run Mode, this page displays the real-time data in graphic format.

- **Configure pDR**: This screen allows the user to edit the operating/logging parameters. Click on the item to be edited and select or type in the new value. To review the parameter values currently programmed into the *personal*DataRAM, click on **Get configuration**. After editing the parameters, click on **Set configuration** to input the new values into the *personal*DataRAM program.

Most operations within *p*DR-COM are self-evidently labeled, including fly-over dialog boxes. In addition, instructions may be found in the On-line Help files by selecting **Help** and then **Contents**.

The following operating/logging parameters of the *personal*DataRAM are selected (edited) via the computer:

- Current date (month and day of the month)
- Current time (hour, minute and second)
- Display averaging time (1 to 60 seconds, in 1-second increments)
- Calibration factor (0.01 to 9.99, in 0.01 increments)
- Logging interval (1 to 14,400 seconds, in 1-second increments)
- Analog output full scale concentration (0.1, 0.4, 1, 4, 10, 40, 100, or 400 mg/m^3)
- Analog output status (enabled, or disabled) (can also be selected directly through *personal*DataRAM keyboard, see Section 8.2)
- Alarm level (0.001 to 409.599 mg/m^3, in 1-µg/m^3 increments)
- Alarm mode (Off, Instantaneous, or STEL) (can also be selected directly through *personal*DataRAM keyboard, see Section 8.2)

The serial number of the *personal*DataRAM is transferred automatically to the PC and displayed on its screen.

In addition, the user can input any other identification for the instrument (up to 20 characters).

Note: The year is entered as a two-digit number; year 2000 is treated correctly as a leap year (*personal*DataRAM version 1.70 or higher).

9.4 Real-Time RS-232 Output

During the RUN mode, the *personal*DataRAM can communicate real-time concentration data through its serial port via the *p*DR-COM software package. This software application decodes the data and displays it on the computer screen in both graphical and tabulated form.

In order to use this output with some other application, the following information will enable the user to decipher the encoded output signal.

The communication settings for the digital output of the *personal*DataRAM are:

- Baud rate: 4800 bps
- Data bits: 8
- Stop bits: 1
- Parity: none
- Flow control: Xon/Xoff

Every second during a run, the *personal*DataRAM serial port will output a sixteen-character code. It consists of two brackets with 14 hexadecimal digits between them,

representing sum check (2 digits), sensed concentration (8 digits), and calibration factor (%, 4 digits). The concentration in $\mu g/m^3$ is obtained by multiplying the sensed concentration times the calibration factor and dividing by 100.

10.0 ANALOG SIGNAL OUTPUT

10.1 Analog Output Description

The *personal*DataRAM incorporates the capability to provide both a voltage and a current signal output directly proportional to the sensed concentration of airborne particulates. Both these analog signal outputs are concurrently available. These outputs are provided, principally, for fixed point applications with hard-wired installations, such as for continuous HVAC monitoring and control.

The particulate concentration range corresponding to the output voltage and current ranges (0 to 5 V and 4 to 20 mA) can be user selected (via a PC). The most sensitive range available is 0 to 0.100 mg/m^3, and the least sensitive range is 0 to 400 mg/m^3. For example, if the user selects the analog output range of 0 to 0.400 mg/m^3 then the analog output signal levels, at a concentration of 0.200 mg/m^3, would be 2.5 V and 12 mA.

Selection of the concentration range of the analog output must be performed on the PC. This range is independent of the digital display, data logging and real-time digital output range which are controlled automatically (auto-ranging).

Enabling the analog output increases the current consumption from the power source (battery or power supply) of the *personal*DataRAM by typically 5 mA when no load is connected to the analog signal current output. If such a load is connected then the current consumption of the *personal*DataRAM further increases by the magnitude of the output signal current (up to a maximum increment of 20 mA). Therefore, when not using the analog output, it is advisable to disable that output (see Section 8.2) in order to minimize power consumption (this is important only when powering the *personal*DataRAM from a battery source).

10.2 Analog Output Connection

The *personal*DataRAM is provided with a cable (model *p*DR-ANC) which has a 6-contact plug at one end and flying leads at the other. There are 4 leads for the analog and alarm outputs. The additional two contacts of the connector are used only for digital communication with a PC, for which a separate cable (model *p*DR-DCC) is provided.

Counting from top to bottom on the *personal*DataRAM connector receptacle, contact #1 is the positive 4 - 20 mA analog output, contact #2 is the alarm output, contact #5 is the common ground (return for all signals), and contact #6 is the positive 0 - 5 V analog output.

For the 0 - 5 V output signal, the externally connected load must have an impedance of more than 200 kilo-ohms. For the 4 - 20 mA output signal, the externally connected load must have an impedance of less than 200 ohms when powering the *personal*DataRAM with a battery, or less than 300 ohms when using the its AC supply.

Since both voltage and current outputs are present at the same time, both can be used concurrently, if so required.

The accuracy of the analog output signals is better than 1% of the reading with respect to the digital reading.

11.0 ALARM

11.1 Alarm Description and Operation

The *personal*DataRAM alarm function is provided both as an audible signal as well as an electrical output. The audible alarm consists of a series of beeps generated by an on-board piezo-transducer. The electrical output, available at the digital communications port, consists of a 1 Hz square wave signal which can be used to trigger/activate other equipment through an appropriate interface (consult with the factory).

The alarm function can be enabled/disabled by the user through the *personal*DataRAM keyboard (see Section 8.2). Setting of the alarm level must be performed on the PC (see Section 9.0).

The alarm is triggered whenever the preset alarm level is exceeded based either on : a) the displayed real-time concentration, if ALARM: INSTANT was selected (see Section 8.2), or b) a 15-minute running average concentration, if ALARM: STEL was selected. When the concentration falls below that level the alarm condition stops. While the alarm is on the user can stop it (i.e. silence the alarm) by pressing any key of the *personal*DataRAM. If the concentration continues to exceed the set alarm level after 10 seconds, however, the alarm restarts.

11.2 Alarm Output

A pulsed voltage output is available on the *personal*DataRAM in synchronism with the audible signal. This signal consists of a 1 Hz square wave with an amplitude of 5 V pp. An externally connected load should have an impedance of no less than 100 kilo-ohms. This alarm output signal is available at pins 2 and 5 (counting from top to bottom) of the 6-contact output/communications port on the side of the *personal*DataRAM (see Figure 5 or 6).

11.3 Remote Alarm Unit

An alarm relay unit (MIE model *p*DR-RA) is available as an optional accessory for the *personal*DataRAM. The *p*DR-RA, when connected to the alarm output of the

*personal*DataRAM, provides a switched output triggered by the alarm signal of the monitor. This switched output (up to 8 amperes, 250 volts) can be used to activate or deactivate other equipment (e.g. ventilation systems, machinery, etc.), or to control remotely located (by wire connection) alarm indicators (e.g. buzzers, lights, etc.).

12.0 MAINTENANCE

12.1 General Guidelines

The *personal*DataRAM is designed to be repaired at the factory. Access to the internal components of the unit by others than authorized MIE personnel voids warranty. The exception to this rule is the occasional cleaning of the optical sensing chamber.

Unless a MALFUNCTION message is displayed, or other operational problems occur, the *personal*DataRAM should be returned to the factory once every two years for routine check out, test, cleaning and calibration check.

12.2 Cleaning of Optical Sensing Chamber

Continued sampling of airborne particles may result in gradual build-up of contamination on the interior surfaces of the sensing chamber components. This may cause an excessive rate of increase in the optical background. If this background level becomes excessive, the *personal*DataRAM will alert the user at the completion of the zeroing sequence, as indicated in Section 8.1, by the display of a BACKGROUND HIGH message. If this message is presented, the *personal*DataRAM can continue to be operated providing accurate measurements. However, it is then advisable to clean the interior of the sensing chamber at the first convenient opportunity, proceeding as indicated below.

12.2.1 Model *p*DR-1000AN

- Remove the two screws on the top of the large protective bumper that covers the sensing chamber (see Figure 1);
- Remove the large protective bumper by lifting it firmly upwards and away from the sensing chamber;
- Remove the socket-head screws on the front and back black covers that were exposed by removal of the large top bumper. Lift away the freed front and back covers of the sensing chamber; set them aside carefully and such that they can be reattached in the same position as they were previously; avoid touching the dull black side of these plates;
- Using filtered (particle-free) pressurized air, blow the inside of the sensing chamber taking great care in not marring or scratching any of the exposed surfaces;
- Reposition the two sensing chamber cover plates in the same location (front and back) as they had been originally. Insert and tighten socket head screws firmly

making sure that the two plates are aligned perfectly with the top of the sensing chamber;
- Reposition large protective bumper over sensing chamber pushing down until properly seated. Insert the two top screws holding down the bumper and tighten gently (do not over-tighten);
- Check optical background by zeroing the *p*DR-1000AN as indicated in Section 8.1. If the sensing chamber cleaning was performed correctly, the message CALIBRATION: OK should be displayed at the end of the zeroing period.

12.2.2 Model *p*DR-1200

- Remove the two screws (one in the front and one in the back) holding the front and back gasketed covering plates of the sensing chamber, and set these plates aside, such that they may be reattached in the same location as they were previously.
- Using filtered (particle-free) pressurized air, blow the inside of sensing chamber taking great care in not marring or scratching any of the exposed surfaces.
- Reposition the two sensing chamber cover plates in the same location (front and back) as they had been originally. Insert and tighten socket head screws firmly making sure that the two plates are aligned perfectly with the top of the sensing chamber.
- Check optical background by zeroing the *p*DR-1200 as indicated in Section 8.1. If the sensing chamber cleaning was performed correctly, the message CALIBRATION: OK should be displayed at the end of the zeroing period.

12.3 Cyclone Cleaning (Model *p*DR-1200 only)

The cyclone will require occasional cleaning. It is advisable to do so whenever the sensing chamber of the *p*DR-1200 is cleaned (see above). To clean the cyclone, remove it from its black attachment cup on the sensing chamber , and unscrew the grit pot (narrower knurled end). Use clean pressurized air to blow out the grit pot and through all openings of cyclone body. Reattach grit pot to cyclone body and insert cyclone body into attachment cup making sure it is fully inserted.

13.0 CALIBRATION

13.1 Factory Calibration

Each *personal*DataRAM is factory calibrated against a set of reference monitors that, in turn, are periodically calibrated against a gravimetric standard traceable to the National Institute of Standards and Testing (NIST).

The primary factory reference method consists of generating a dust aerosol by means of a fluidized bed generator, and injecting continuously the dust into a mixing chamber from which samples are extracted concurrently by two reference filter collectors and by two master real-time monitors (MIE DataRAMs) that are used for the routine calibration of every *personal*DataRAM.

The primary dust concentration reference value is obtained from the weight increase of the two filters due to the dust collected over a measured period of time, at a constant and known flow rate. The two master real-time monitors are then adjusted to agree with the reference mass concentration value (obtained from averaging the measurements of the two gravimetric filters) to within ±1%.

Three primary, NIST traceable, measurements are involved in the determination of the reference mass concentration: the weight increment from the dust collected on the filter, the sampling flowrate, and the sampling time. Additional conditions that must be met are: a) suspended dust concentration uniformity at all sampling inlets of the mixing chamber; b) identical sample transport configurations leading to reference and instrument under calibration; and c) essentially 100% collection efficiency of filters used for gravimetric reference for the particle size range of the test dust.

The test dust used for the MIE factory calibration of the *personal*DataRAM is SAE Fine (ISO Fine) supplied by Powder Technology, Inc. It has the following physical characteristics (as dispersed into the mixing chamber):

- Mass median aerodynamic particle diameter: 2 to 3 μm
- Geometric standard deviation of lognormal size distribution: 2.5
- Bulk density: 2.60 to 2.65 g/cm^3
- Refractive index: 1.54

13.2 Field Gravimetric Calibration

If desired, the *personal*DataRAM can be calibrated gravimetrically for a particular aerosol (dust, smoke, mist, etc.) under field conditions (actual conditions of use). To effect such calibration in the particle environment of interest, proceed as indicated below.

For field calibration of the model *p*DR-1000AN, a personal type filter sampler is placed side-by-side (collocated) to the *p*DR-1000AN to be calibrated, and the two units should be started simultaneously. For the model *p*DR-1200, its own filter and attached pump can be conveniently used for the same purpose.

- Weigh and load into filter holder a fresh membrane filter.
- Start pump.
- Immediately turn on *personal*DataRAM and start a run such that the pump and the *personal*DataRAM are started nearly simultaneously.

The duration of this comparison run should be sufficient to collect a mass of at least 1 mg on the reference filter (in order to permit accurate weighing of the collected mass by means of an analytical balance). The time-weighted average (TWA) reading of the *personal*DataRAM can be used to estimate the required sampling time to collect the above-mentioned mass on the filter. To estimate the required sampling time (ET as measured on the *personal*DataRAM) in minutes, read the TWA value

(see Section 8.3) after an elapsed time (ET) of one minute or more, and apply the following relationship:

$$ET \geq 500/TWA$$

For example, if TWA = 2.5 mg/m^3, then ET ≥ 200 minutes (approximately 3 hours). If the TWA value changes significantly as the run proceeds, recalculate the required ET accordingly.

At the end of the run (after time ET has elapsed), record TWA, ET and the flow rate Q used to sample the air. Weigh the filter on an analytical balance and obtain Δm, the mass increment due to the collected particles.

Calculate the average gravimetric concentration C, as follows:

$$C = 1000\ \Delta m/ETxQ$$

Compare the recorded value of TWA and the calculated value C, and calculate the calibration factor to be programmed into the *personal*DataRAM (see Section 9.0) as follows:

$$CAL\ FACTOR = C/TWA$$

For example, if C was found to be 3.2 mg/m^3, and TWA had been determined to be 2.5 mg/m^3, the CAL FACTOR equals 1.28. Select this value on the PC, as described in Section 9.0. This completes the gravimetric calibration of the *personal*DataRAM for a specific aerosol.

13.3 Scattering Coefficient Calibration

Users interested in using the *personal*DataRAM for scattering coefficient measurements (e.g., for atmospheric visibility monitoring) should contact the factory. A special primary Rayleigh scattering calibration for such purpose can be performed by MIE.

13.4 Internal Span Check

The zeroing procedure (see Section 8.1) and the resulting normal diagnostic display of "CALIBRATION: OK" (step 2) informs the user that the instrument's calibration agrees with the original factory setting. This is an internal span check that consists of an automatic comparison between the initial (factory) optical background of the *personal*DataRAM (registered in its non-volatile memory), and the current optical background sensed during the zeroing sequence.

14.0 PARTICLE SIZE CLASSIFICATION (model *p*DR-1200 only)

The particle size selective cyclone of the *p*DR-1200 provides the user with two important capabilities: a) to measure the particulate matter concentration of a specific aerodynamic size fraction, and b) to determine the mass median size of a particle population. These two applications will be discussed in what follows. For both these applications, a variable measured flow rate pump is required, such as the MIE model *p*DR-PU (for which a separate instruction manual is provided).

14.1 Size Fractionated Monitoring

The *p*DR-1200 can be used to monitor a specific particle size fraction below a selectable cut off equivalent aerodynamic diameter. The particle size cut point can be selected by adjustment of the sampling flow rate. The higher this flow rate through the cyclone the smaller the cut off particle diameter. Figure 7 is a graph showing the dependence of the particle cut off size in micrometers as a function of the sampling flow rate in liters per minute. The cut off size is the particle aerodynamic diameter at which the collection efficiency of the cyclone is 50%, or conversely, the size at which the cyclone transmission is 50%. For example, to obtain a particle size cut off of 2.5 μm (i.e., $PM_{2.5}$), the required sampling flow rate is 4 liters/minute. A that flow rate only particles smaller than (approximately) 2.5 μm are allowed to pass into the *p*DR-1200 sensing stage, to be monitored and then to be collected on the filter.

As can be seen on Fig. 7, the lowest particle size cut for the GK 2.05 cyclone included with the *p*DR-1200 is about 1 μm, and the largest is about 12 μm. For particle size classification outside this range, consult with MIE.

14.2 Particle Sizing

The selectable particle size capability of the cyclone, in combination with the concentration measuring capability of the photometric system of the *p*DR-1200 permits the user to determine the mass median aerodynamic particle diameter of an aerosol, i.e., of the airborne particle population being sampled.

One simple procedure to determine the median particle size is as follows (please refer to the graph of Fig. 7):

- Remove cyclone from its black attachment cup and set cyclone aside
- Start pump and sample aerosol at a flow rate between 2 and 4 liters/minute
- Press **ON** key on *p*DR-1200 panel and after about one minute key **NEXT** and then **ENTER**
- After an elapsed time (ET) of about one minute, read and note TWA concentration
- Shut off pump

- Plug in cyclone into its attachment cup

- Start pump and run at about 1 liter/minute. Observe real-time concentration (CONC) reading

- Increase flow rate very slowly and gradually until CONC reading is one-half of the initial concentration measured without the cyclone. Continue sampling at this
  flow rate for about one minute and confirm that TWA reading is about one-half of the initial one. Otherwise readjust flow rate. Note final flow rate at which the TWA value has decreased to one-half the value noted without the cyclone.

- Enter the final flow rate for which the TWA value is one-half of the initial value into the graph of Fig. 7 and read the corresponding d_{50} particle size in micrometers. This represents the mass median particle diameter of the aerosol.

For example, if the TWA value without the cyclone was 0.8 mg/m³, and the flow rate (with the cyclone attached) required to reduce the TWA to 0.4 mg/m³ is 2 liters/minute, the mass median particle size (as obtained from the curve of Fig. 7) is approximately 5.5 μm.

15.0 CONVERSION BETWEEN *personal*DataRAM VERSIONS

The *personal*DataRAM user has the option to convert from a model *p*DR-1000AN to a model*p*DR-1200 or vice versa using the appropriate conversion kit. To convert from a *p*DR-1000AN to a *p*DR-1200 (i.e., from a passive air sampling configuration to an active one), the user requires the model *p*DR-ASC conversion kit. To convert from a *p*DR-1200 to a *p*DR-1000AN (i.e., from an active air sampling configuration to a passive one), the user requires the model *p*DR-UB conversion kit.

15.1 Conversion Procedure From *p*DR-1000AN to *p*DR-1200

To effect this conversion use model *p*DR-ASC conversion kit. As you remove parts from the *p*DR-1000AN, in order to attach the conversion kit components, store these parts carefully for possible future re-conversion. Proceed as follows:

- Remove the two screws on the top of the large protective bumper that covers the sensing chamber (see Figure 1). This bumper is not used on the *p*DR-1200;
- Remove the large protective bumper by lifting it firmly upwards and away from the sensing chamber;
- Reinsert in the upper two threaded holes and tighten the two screws that had held the protective bumper;
- Remove the socket-head screws on the front and back black covers that were exposed by removal of the large top bumper. Lift away the freed front and back covers of the sensing chamber; store them carefully for future use, ensuring that their surfaces are not scratched or marred;

- Position one of the two gasketed (soft rubber) sensing chamber cover plates provided in the conversion kit on the front side of the sensing chamber. Insert and tighten the included socket head screw firmly making sure that the plate is aligned perfectly with the top of the sensing chamber. Similarly, attach the other cover plate on the back side of the sensing chamber;
- Identify the two black cups of the *p*DR-ASC conversion kit. One of them has an external o-ring (filter holder cup), and the other has no o-ring (cyclone cup); refer to Figures 2 and 4 for the location of these cups on the *p*DR-1200 sensing chamber. These cups can be installed on either side of the sensing chamber, i.e., the cyclone can be either on the left or the right side of the sensing chamber (Figure 2 shows the case where the cyclone is on the right side);
- Attach one cup to the left side of the sensing chamber using the two black socket head screws. Tighten screws firmly. Similarly, attach the other cup to the right side of the sensing chamber;
- Take the cyclone/filter holder unit provided as part of the conversion kit, and separate the 37-mm plastic filter holder from the metal cyclone by firmly pulling the two units apart;
- Carefully slide the large open end of the plastic filter holder over the cup with the external o-ring, previously attached to the sensing chamber. Ensure that the cup is fully inserted into the filter holder;
- Carefully insert the large diameter open end of the metal cyclone into the other cup on the opposite side of the sensing chamber. The cyclone inlet (small short metal tube on side of cyclone) can be oriented as desired (upwards, as shown in Figure 2, sideways, downwards, etc.). Ensure that the cyclone is fully inserted into the cup;
- When ready to operate, connect a length of tubing between the barbed fitting at the downstream end of the plastic filter holder and the pump to be used in combination with the *p*DR-1200.
- Perform a zeroing sequence (see Sections 6.5.2 and 8.1) before starting a run. This completes the conversion of the *p*DR-1000AN to the *p*DR-1200.

15.2 Conversion Procedure from *p*DR-1200 to *p*DR-1000AN

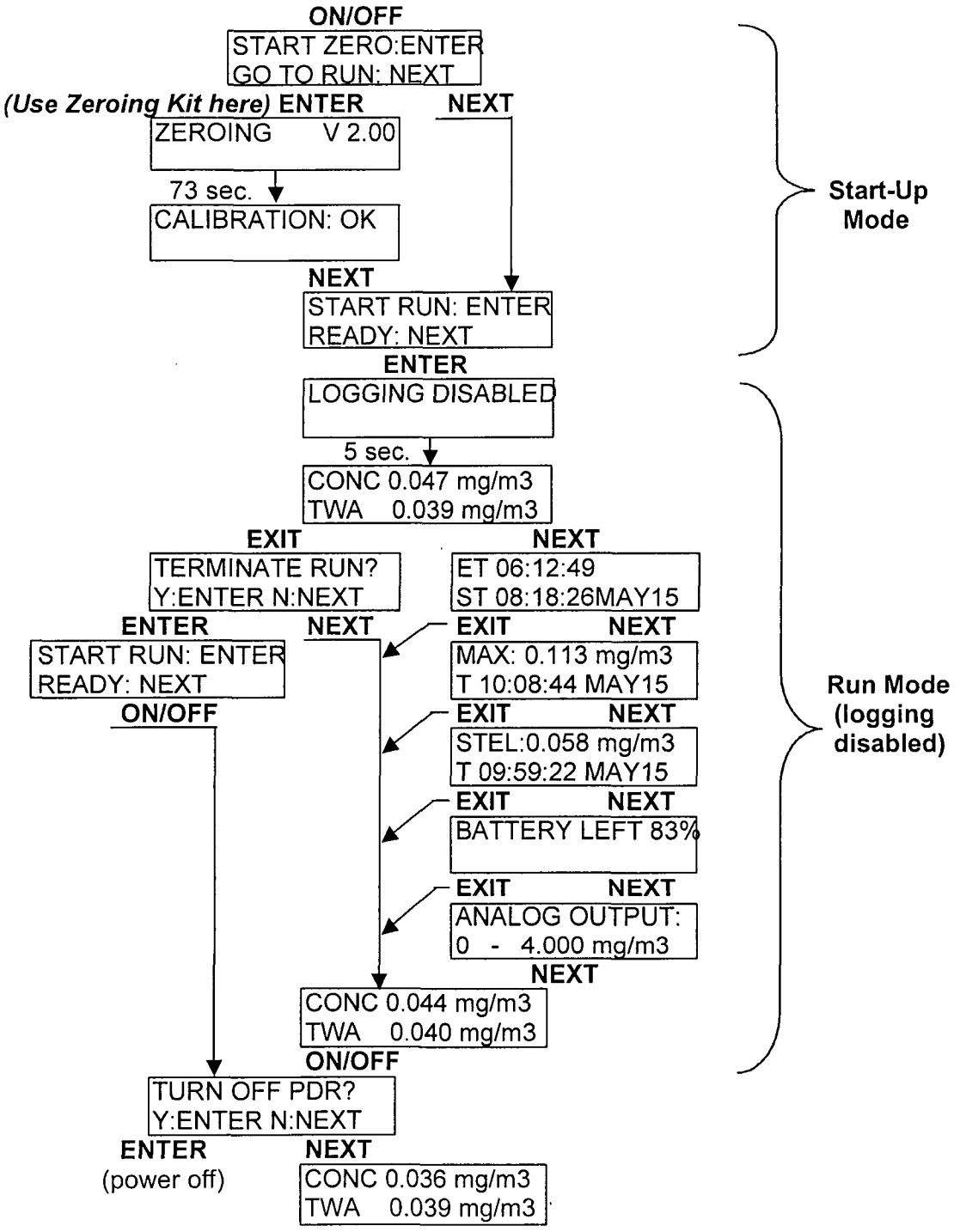
To effect this conversion use model *p*DR-UB conversion kit. As you remove parts from the *p*DR-1200, in order to attach the conversion kit components, store these parts carefully for possible future re-conversion. Proceed as follows:

- Pull off both the cyclone and the filter holder from their respective cups on the two sides of the sensing chamber;
- Loosen the two screws that hold each of the two cups on the sides of the sensing chamber (total of 4 screws), and remove the two side cups;
- Loosen the single screw on each of the two (front and back) gasketed sealing covers enclosing the sensing chamber, and remove the two covers;
- Identify the two flat sensing chamber cover plates provided in the conversion kit; one face of each of each of these two plates has a dull black finish (antireflective); avoid touching those surfaces;

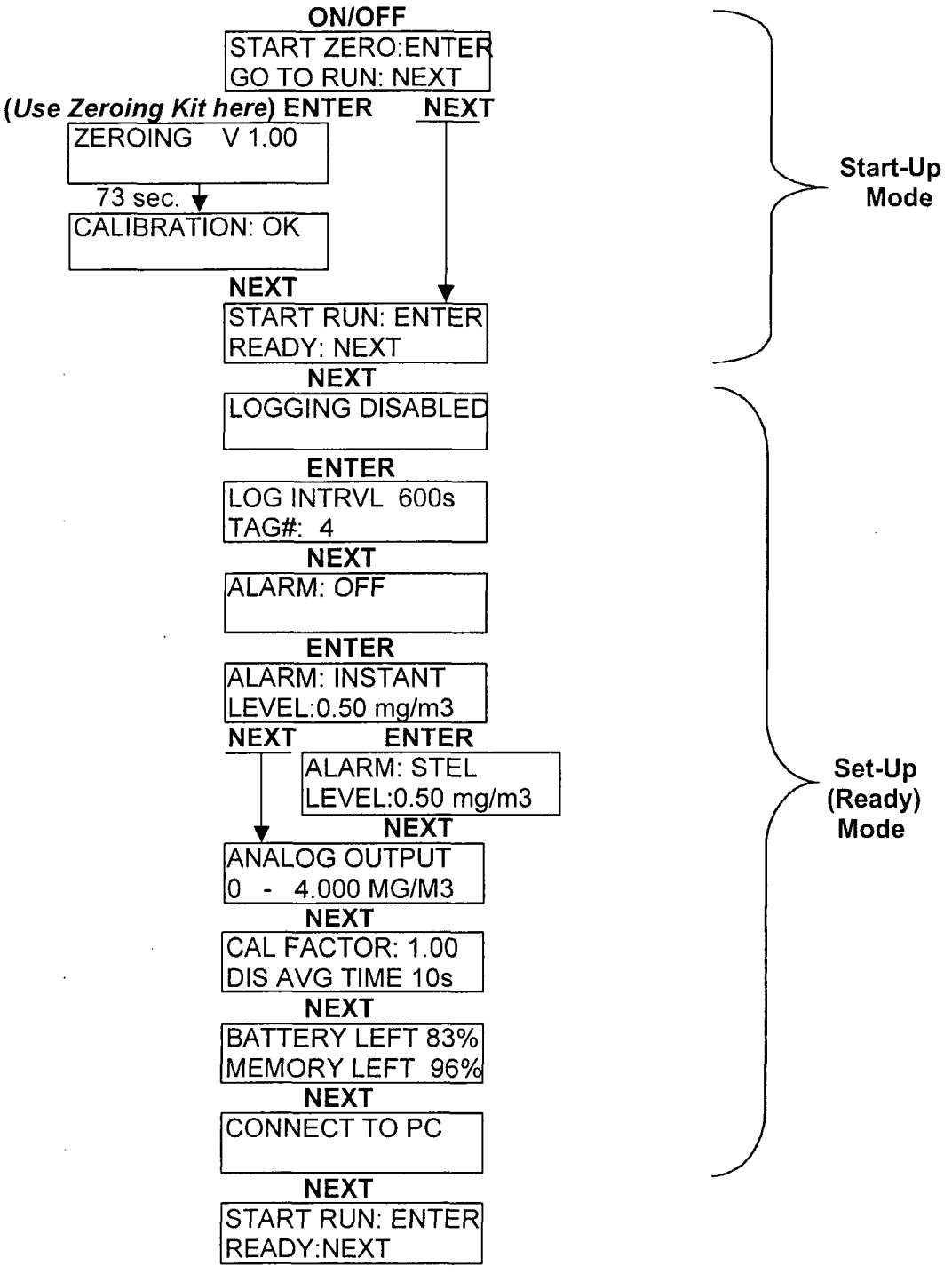
- Position one of the two sensing chamber cover plates over the open front of the sensing chamber with the dull surface on the inside, and such that the hole in the plate is aligned with the corresponding threaded mounting hole on the upper wall of the sensing chamber. Insert and tighten firmly black socket head screw provided with the conversion kit, making sure that the plate is aligned perfectly with the top of the sensing chamber. Similarly, attach the other cover plate to the rear of the sensing chamber, with the dull surface facing inward;
- Loosen and remove the two small screws on the top surface of the sensing chamber;
- Position large protective bumper (provided in the conversion kit) over sensing chamber pushing down until properly seated. Insert the two top screws (two shiny Phillips-head screws provided in the conversion kit) Into the two holes In the bumper while holding down the bumper, and tighten gently (do not over-tighten) making sure that the heads of these screws are well Inside their cavities In the bumper;
- Perform a zeroing sequence (see Sections 6.5.1 and 8.1) before starting a run. This completes the conversion from a *p*DR-1200 to a *p*DR-1000AN.

16.0 SEQUENCE OF KEYSTROKES AND SCREENS (*p*DR-1000 AN, -1200, HPM-1000)

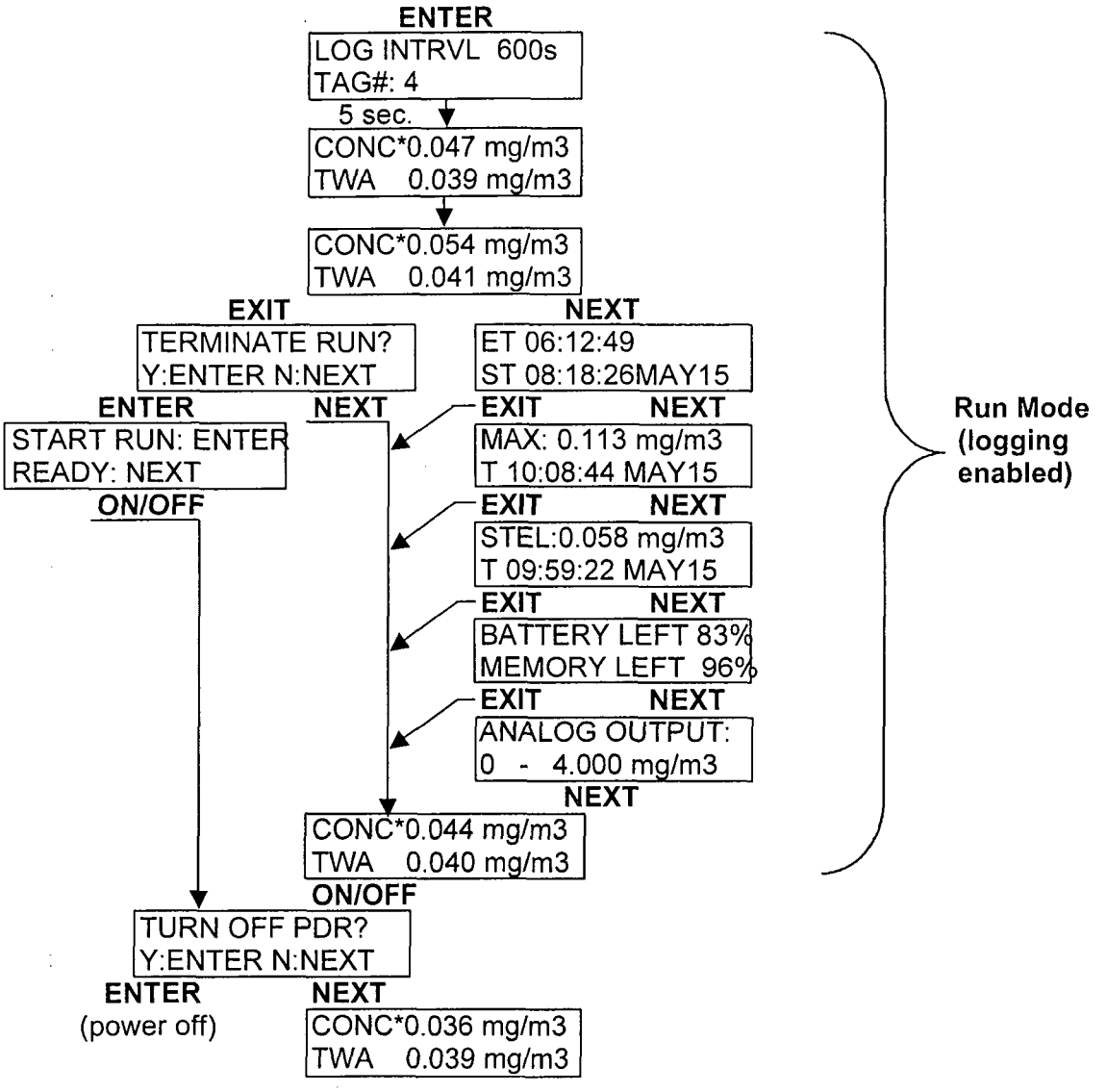
Start-Up and Survey Run Mode (*Without* Data Logging)



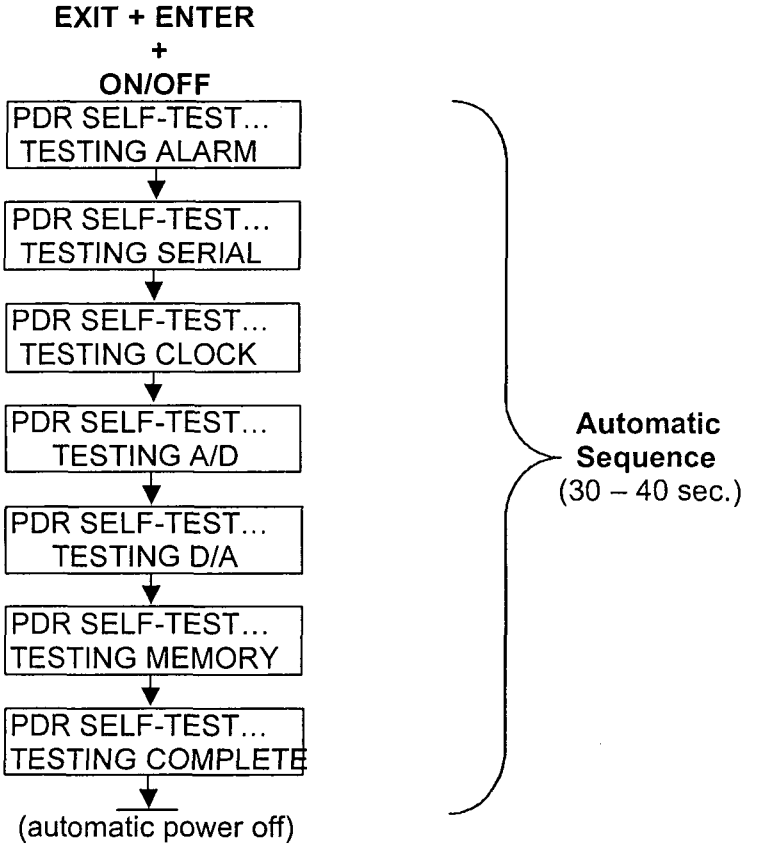
Start-Up, Set-Up and Run Mode (*With* Data Logging)



(Continues on next page)



Resetting/Electronic Checking Mode



NOTE: After the preceding resetting sequence, the instrument should be zeroed, otherwise its optical background will remain unsubtracted.

STL-Edison Standard Operating Procedure	Revision Date: 03/09/2004
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1. SCOPE and APPLICATION

1.1. This procedure explains the process of review of Quality Control indicators that are performed in accordance with the methodology being employed.

1.2. The specific criteria that are evaluated are method specific and are enumerated in the respective analytical SOPs. Detailed audit procedures including NJDEP QA requirements for each method are given in the following QA Checklists:

Attachment	QA Checklist Title
1	Volatile Organics, SW846 Method 8260B
2	Semivolatile Organics, SW846 Method 8270C
3	Organochlorine Pesticides and PCBs, SW846 Method 8081A/8082
4	Metals by ICP, SW846 Method 6010B
5	Metals by Graphite Furnace AA, SW846 7000 Series Methods
6	Mercury by Cold Vapor AA, SW846 Method 7471A
7	General Chemistry
8	Volatile Organics, USEPA SOW
9	Semivolatile Organics, USEPA SOW
10	Metals by ICP, USEPA SOW

11 Metals by Graphite Furnace AA, USEPA SOW

12 Mercury by Cold Vapor AA, USEPA SOW

1.3. Corrective action procedures are presented in Section 3 of this SOP as guidance when criteria described in Attachments 1 through 12 are out of acceptance limits.

2. PROCEDURE

2.1. The ultimate responsibility for quality inspection rests with the Quality Assurance Manager. The review of Quality Control criteria is performed in a tiered approach. The first level of review is performed by the analyst conducting the test, the next tier of review rests with the department manager and the final tier of review is performed by the Laboratory Manager.

2.2. Environmental samples are analyzed by different methodologies within a department. The analyst determines which methodology is to be followed before initiating the analysis. Instrument tunes and calibrations are conducted in accordance with the method. If any criteria do not completely satisfy the method requirements for tune and calibration, the analyst stops and rectifies the problem in accordance with the specific analytical SOP. The analyst may seek the assistance of the supervisor.

2.3. The root cause for a nonconformance to method criteria may either be local or systematic. A local nonconformance requires a solution that is isolated to a particular instrument and is readily rectified. A systematic nonconformance is normally present across all instruments in a department. Local nonconformance may be rectified by the analyst alone or by the analyst and the department manager. If at any time a systematic nonconformance is suspected, the Quality Assurance Manager is notified and the QAO, department manager and one or more analysts initiate a corrective action investigation to isolate and eliminate the root cause of the nonconformance.

2.4. Analysis of samples may occur only after the cause of the nonconformance has been eliminated. The analyst proceeds to acquire data in accordance with the method being followed. After an analytical sequence is complete, each sample is reviewed to determine if it meets the method criteria set forth in the analytical SOP. Specific criteria may vary from method to method and include but are not limited to analytical acquisition occurring within a specified clock, areas of internal standards, surrogate recoveries within acceptable limits, post digestate spike recoveries within limits and absence of contamination in the laboratory blank.

2.5. Particular attention is paid to the Quality Control samples that were run. These samples are subject to the same criteria as typical samples but also give indications of local or systematic nonconformance. An analyst need not notify the department manager if the laboratory blank, Matrix Spike, Matrix Spike Duplicate and Blank Spike samples are within method specifications or within the laboratory's control limits as established by Control Charts. The method being employed determines whether laboratory established or method established criteria are to be used. If any of these samples are outside acceptable limits the department manager is notified and reporting of all samples associated with the QC samples is withheld pending a determination as to the cause of the nonconformance.

2.6. Whenever a nonconformance is reported, the supervisor collects all relevant information relating to the analysis in order to follow a logical path to sound judgment. The supervisor first determines if the nonconformance is local or systematic. If a systematic nonconformance is suspected, the Quality Assurance Manager is notified and a corrective action investigation is initiated. Typically, samples will be re-extracted and rerun if sufficient sample exists to confirm local nonconformance. The Quality Assurance Manager has the final authority and say if there is a disagreement.

2.7. The correction of a local nonconformance is documented in the analytical run logbook.

2.8. All instances of nonconformance are explained on the jobs Non-Conformance Summary that is kept in the job specific folder in the Data Management Office.

2.9. Periodically, random, unannounced comprehensive QA audits are conducted by the Quality Assurance Manager.

3. CORRECTIVE ACTIONS

3.1. Corrective Actions are taken based upon QA Checklists presented in Attachments 1 through 12. The type of action that may be taken is based upon an overall assessment of factors relating to a particular testing program and when a problem is discovered (i.e. is the sample still within its holding time).

3.2. The corrective action most frequently required when a question in Attachments 1 through 12 is answered "no" is to reject the results and re-analyze the samples. This assumes that the specific problems noted in the QA Checklist is discovered when the sample is within holding time. Accordingly the first round of QA review is performed by the analyst conducting the test and resolved with their supervisor.

3.3. Corrective action taken if it is discovered that a sample exceeds holding time includes contacting the client to determine if new samples that are within holding time are available. If new samples are available perform analysis within holding time, if not note holding time nonconformance in the case narrative or nonconformance summary.

ATTACHMENT 1
QUALITY CONTROL CHECKLIST
SW-846, METHOD 8260B, VOLATILE ORGANIC COMPOUNDS BY GC/MS

		Yes	No
I.	I. Sample Preservation, Storage & Holding Times.		
	A. Were soil samples analyzed within 14 days of collection?	____	____
	B. Were water samples preserved and analyzed within 14 days of collection?	____	____
	C. Were all samples stored at 4°C in darkness until analyzed?	____	____
	D. If Method 5035 Prep was employed were the Field cores preserved within 48 hours with Sodium Bisulfate solution or by freezing?	____	____
II.	Sample Preparation & Cleanup		
	A. Were reference standards for spike solutions obtained from independent lots?	____	____
	B. Did the purge and trap device agitate all low level soil samples during the purge step as required?	____	____
	C. Were all low level soil sample vials pre-preserved with Sodium bisulfate solution?	____	____
	D. Were all high level soil samples vials pre-preserved with methanol?	____	____
	E. Were all pre-preserved soil sample vials pre-weighed in the laboratory before shipment to the field?	____	____
	F. If methanol preservation was employed for high concentration soil analysis, was a methanol blank successfully analyzed which met all blank criteria?	____	____
	G. If Sodium bisulfate preservation was employed for low concentration soil analysis, was a sodium bisulfate blank successfully analyzed which met all blank criteria?	____	____
III.	Instrument Tuning & Initial Calibration.		
	A. Was every analytical sequence initiated by injecting 50 ng of BFB which met the method specified mass and abundance criteria?	____	____
	B. Was the required background subtraction for BFB employed by selecting a scan for background subtraction $\leq$ 20 scans prior to the elution of BFB?	____	____
	C. Were the standard or standards run after the injection of BFB?	____	____
	C. Was an initial 5 point calibration of all target analytes performed?	____	____
	E. Were separate initial calibrations prepared for each sample introduction technique?	____	____

	Yes	No
F. Was internal standard added to each calibration standard to achieve a final concentration of 50 ug/l?	____	____
G. Was sodium bisulfate preservative solution included as required in the calibration standards used for low level soil analysis?	____	____
H. Was the relative retention time (RRT) of each compound within 0.8 - 1.2 RRT units of its assigned internal standard?	____	____
I. Did the relative retention times of each compound in the five calibration standards agree within 0.06 relative retention times units?	____	____
J. Were the average RFs of the five system performance check compounds (SPCC) $\geq$ the following criteria?		
1. Chloromethane 0.10	____	____
2. 1,1-Dichloroethane 0.10	____	____
3. Bromoform 0.10	____	____
4. Chlorobenzene 0.30	____	____
5. 1,1,2,2-Tetrachloroethane 0.30	____	____
K. Was the RSD of the 6 calibration check compounds (CCC) $\leq$ 30%	____	____
L. For initial calibration, one of three approaches may be taken in the following order of preference:		
1. A linear 5 point calibration using the average calibration factor:		
(a) Was an initial 5 point calibration for all single component analytes and surrogates analyzed, and was the %RSD $\leq$ 15% for each analyte?	____	____
(b) If one or more compounds have %RSD > 15%, was the average %RSD for <u>all</u> analytes $\leq$ 15%? (If so, the average calibration factor may be used provided the data user is provided with a summary of all analytes whose %RSD exceeded 15%).	____	____
M. Were surrogate compounds added to each of the standards?	____	____
N. Was purge heated to 40°C for low level analysis?	____	____

IV. Calibration Verification

A. Was the calibration verified every 12 hours as required?	____	____
B. Did the verification begin with an injection of 50 ng of BFB that met the method specified criteria?	____	____
C. Was the required background subtraction for BFB performed selecting a scan for background subtraction $\leq$ 20 scans prior to the elution of BFB?	____	____
D. Was the calibration verification standard run immediately after or along with the BFB?	____	____
E. Were all target analytes injected for continuing calibration		

	Yes	No
verification?	____	____
F. Were the following internal standards spiked into all standard solutions to achieve a final concentration of 50 ug/l?	____	____
1. Fluorobenzene	____	____
2. Chlorobenzene – d5	____	____
3. 1,4-Dichlorobenzene-d4	____	____
G. Was the concentration of the calibration check standard at the midpoint of the calibration range?	____	____
H. Were the RFs of the five system performance check compounds (SPCC) ≥ the following criteria?		
1. Chloromethane 0.10	____	____
2. 1,1-Dichloroethane 0.10	____	____
3. Bromoform 0.10	____	____
4. Chlorobenzene 0.30	____	____
5. 1,1,2,2-tetrachloroethane 0.30	____	____
I. Was the percent difference of the 6 calibration check compounds (CCC) ≤20%?	____	____
J. If CCCs were not among the project analytes, did all target analytes meet the 20%D or 20% drift criteria?	____	____
K. Were the retention times of the internal standards from the calibration check within ±30 seconds of the internal standards from the mid point standard of the original calibration?	____	____
L. Was the response (area count) of each internal standard in the calibration verification standard within a factor of 2 (-50% to + 100%) of its corresponding internal standard in the mid-level calibration standard of the active calibraton curve?	____	____
M. Did the relative retention times of each compound in the calibration verification standard agree within 0.06 relative retention time units of its value in the initial calibration?	____	____
N. Were the average response factors from the original five point calibration used for quantitative analysis?	____	____
O. Was the calibration verified for all target compounds every 12 hours? One of the two following options result in a successful calibration verification.	____	____
1. For a linear initial calibration using average calibration factors, calculate the % Difference (%D) of the calibration verification. Do all analytes have %D ≤+/-15%? (For non-linear or linear calibrations with non-zero y-intercepts, use % Drift of the calibration verification as the parameter).	____	____
2. If all analytes %D (or % Drift) were not ≤ +/-15% was the average of all %D's (or % Drift's) ≤ 15%? (If this option is chosen, all analytes in the calibration mix must be included and the data user must be provided with a summary of all analytes whose %D > 15%).	____	____

	Yes	No
P. Was a satisfactory method blank analyzed after the calibration check standard?	____	____

V. Sample Analysis.

A. Were all subsequent samples injected within 12 hours of the BFB injection time?	____	____
B. Were the following internal standards spiked into all samples, blanks and QA samples to achieve a final concentration of 50 ug/l?		
1. Fluorobenzene	____	____
2. Chlorobenzene – d5	____	____
3. 1,4- Dichlorobenzene-d4	____	____
C. Was the response (area count) of each internal standard in the sample within a factor of 2 (-50% to + 100%) of its corresponding internal standard in the mid-level calibration standard of the active calibration curve?	____	____
D. Was each sample screened by GC/FID to determine the appropriate dilution prior to GC/MS analysis?	____	____
E. Was the sample diluted and re-analyzed for all analytes whose quantitation ion abundance exceeded the calibration range?	____	____
F. Was the relative retention times of each detected compound within 0.06 relative retention time units of its corresponding compound in the initial calibration or calibration verification?	____	____

VI. Quality Control Parameters

A. Was an Initial Demonstration of Capability for each sample preparation method employed and is the data available for review?	____	____
B. Has method detection limit (MDL) data been developed for each matrix?	____	____
C. Was a method blank analyzed every 12 hours of sample analysis?	____	____
D. Was the method blank analyzed immediately after the calibration standard(s)?	____	____
E. Did the method blank contain less than the MDL of all target compounds?	____	____
F. Were the following surrogate compounds spiked into all samples, blanks and QA samples?		
1. Toluene-d8	____	____
2. Bromofluorobenzene	____	____
3. 1,2- Dichloroethane-d_4	____	____
G. Were the surrogate recoveries within laboratory control limits?	____	____
H. Were samples that did not meet surrogate accuracy criteria re-analyzed as required?	____	____

	Yes	No
I. Was a laboratory control sample (LCS) and a matrix spike/ matrix spike duplicate (MS/MSD) pair analyzed for each group of no more than 20 environmental samples of a specific matrix within a 14 day period?	____	____
J. Did the LCS and MS/MSD contain at a minimum the following compounds at the following concentrations?		
1. 1,1-Dichloroethene - 50ug/l	____	____
2. Trichloroethene - 50ug/l	____	____
3. Benzene - 50ug/l	____	____
4. Toluene - 50ug/l	____	____
5. Chlorobenzene - 50ug/l	____	____
K. Was MS/MSD and LCS accuracy criteria achieved?	____	____
L. Are in-house performance limits used for determining if LCS, MS/MSD, and surrogate accuracy criteria has been achieved?	____	____

VII. Qualitative & Quantitative Identification

A. Are the chromatograms adequately resolved, not overloaded and free of carryover?	____	____
B. Were RT (0.06 RRT units) identification criteria met for all reported compounds?	____	____
C. Were mass spectral identification criteria met for all reported compounds?	____	____
D. Were all detected analytes within the calibration range of the instrument?	____	____
E. Have all calculations involving dilutions been checked?	____	____

VIII. Data Reporting

A. Was the reporting limit based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture? (No analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).	____	____

Attachment 2
QUALITY CONTROL CHECKLIST
SW-846, METHOD 8270C, SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

	Yes	No
I. Sample Preservation, Storage & Holding Times.		
A. Were soil samples extracted within 14 days of collection, was analysis performed within 40 days of extraction?	____	____
B. Were water samples extracted within 7 days of collection, was analysis performed within 40 days of extraction?	____	____
C. Were all extracts stored at 4°C in darkness until analyzed?	____	____
II. Sample Preparation and Cleanup		
A. Were reference standards for spike solutions obtained from independent lots?	____	____
B. Were all QC samples (i.e. MS/MSD/LCS) extracted with their corresponding field sample?	____	____
C. Were all QC samples and their corresponding field sample processed through the same cleanup procedures?	____	____
D. Was adequate recovery demonstrated for each cleanup technique employed for the analysis prior to its use?	____	____
III. Instrument Tuning and Initial Calibration.		
A. Did every analytical sequence commence with an injection of 50 ng of DFTPP that met the method specified mass and abundance criteria?	____	____
B. Was three scan averaging used to determine the base DFTPP scan which consisted of the scan at the peak apex, +1 scan from the apex and –1 scan?	____	____
C. Was the required background subtraction for DFTPP executed by selecting a scan for subtraction ≤20 scans before the elution of DFTPP?	____	____
D. Did the tuning compound solution contain 50ng/ul of 4,4' DDT, pentachlorophenol, and benzidine for column performance assessments?	____	____
E. Was column performance assessment criteria achieved (DDT breakdown ≤20%, normal response for benzidine & penta-chlorophenol with peak tailing <4)?	____	____
F. Were the standard or standards run immediately after the DFTTP?	____	____
G. Was an initial 5 point calibration of all target anal;ytes performed?	____	____
H. Was internal standard added to each calibration standard at a concentration of 40 ug/ml?	____	____
I. Was the relative retention time (RRT) of each compound within 0.8-		

	Yes	No
1.2 RRT units of its assigned internal standard?	____	____
J. Did the relative retention times of each compound in the five calibration standards agree within 0.06 relative retention time units?	____	____
K. Were the average RFs of the four system performance check compounds (SPCC) $\geq$ 0.05?	____	____
L. Was the %RSD of the 13 calibration check compounds (CCC) $\leq$ 30%?	____	____
M. Were surrogate compounds added to all the standards?	____	____
N. For initial calibration, one of three approaches may be taken in the following order of preference:		
1. A linear 5 point calibration using the average response factor:		
a) Was an initial 5 point calibration for all analytes and surrogates analyzed and was the %RSD $\leq$ 15%?	____	____
b) If one or more compounds have %RSD > 15%, was the average %RSD for <u>all</u> analytes $\leq$15%? (If so, the average response factor may be used provided the data user is provided with a summary of all analytes whose %RSD exceeded 15%).	____	____
2. A linear calibration using a least square regression. (This approach is to be used if the first option is not feasible because of prior knowledge of MS response or if an analyte's response always produces a %RSD 15%. It results in a linear relationship that has a non-zero-y-intercept).		
a) Was the origin not forced and not included in the regression?	____	____
b) Was the correlation coefficient, r^1 greater than or equal to 0.99?	____	____
3. A non linear calibration. (This option may be used when a detector's response is known to be non-linear or when the first two options never meet criteria. It is NOT to be used for detectors that have previously demonstrated a linear response, to compensate for detector satuation or to avoid proper instrument maintenance).		
a) Was the origin not forced and not included in the curve fitting algorithm?	____	____
b) Is the calibration curve continuous and does it have a tangent line whose slope is always positive or always negative?	____	____

	Yes	No
c) Is the function no higher than a third order relationship? (i.e. $ax^3 + bx^2 + cx + d$)?	____	____
d) Was an additional standard run for each increasing order of the equation? (i.e. a second order function requires a 6 point calibration and a third order function requires a 7 point calibration).	____	____
O. Is the Coefficient of Determination (COD) of the equation $\geq$0.99 for each analyte?	____	____

IV. Calibration Verification

A. Was the calibration verified every 12 hours as required?	____	____
B. Did the verification begin with an injection of 50ng of DFTPP that met the method specified criteria?	____	____
C. Was the calibration verification standard run immediately after or or along with the DFTPP?	____	____
D. Were all target analytes injected for continuing calibration verification?	____	____
E. Was the concentration of the calibration check standard at the midpoint of the calibration range?	____	____
F. Were the RFs of the four system performance check compounds (SPCC) $\geq$ 0.05?	____	____
G. Was the percent difference (%D) or percent drift of the 13 calibration check compounds (CCC) $\leq$20%?	____	____
H. If CCCs were not among the project analytes, did all target analytes meet the 20% D or 20% drift criteria?	____	____
I. Were the retention times of the internal standards from the calibration check within $\pm$ 30 seconds of the internal standards from the midpoint standard of the original calibration?	____	____
J. Was the response (area count) of each internal standard in the calibration verification standard within a factor of 2(-50% to +100%) of its corresponding internal standard in the mid-level calibration standard of the active calibration curve?	____	____
K. Did the relative retention times of each compound in the calibration verification standard agree within 0.06 relative retention time units of its value in the initial calibration?	____	____
L. Was the calibration verified for all target compounds every 12 hours? One of the two following options result in a successful calibration verification:		
1. For a linear initial calibration using average calibration factors, calculate the % Difference (%D) of the calibration verification. Do all analytes have %D < +/- 15%? (For non-linear or linear calibrations with non-zero y-intercepts, use %Drift of the calibration verification as the parameter).	____	____

		Yes	No
2.	If all analytes %D (or %Drift) were not ≤ +/- 15% was the average of all %D's (or % Drift's) ≤ 15%? (If this option is chosen, all analytes in the calibration mix must be included and the data user must be provided with a summary of all analytes whose %D > 15%.	____	____

V. Sample Analysis.

		Yes	No
A.	Were all subsequent samples injected within 12 hours of the DFTPP injection time?	____	____
B.	Were the following internal standards spiked into all samples, blanks and QA samples at the concentrations indicated?		
	1. 1,4-Dichlorobenzene-d_4 40ug/ml?	____	____
	2. Naphthalened_8 40ug/ml?	____	____
	3. Chrysened_{12} 40ug/ml	____	____
	4. Acenapthened_{10} 40ug/ml	____	____
	5. Phenanthrened_{10} 40ug/ml	____	____
	6. Perylened_{12} 40ug/ml	____	____
C.	Was the response (area count) of each internal standard in the sample within a factor of 2(-50% to + 100%) of its corresponding internal standard in the mid-level calibration standard of the calibration curve?	____	____
D.	Was each sample screened by GC/FID to determine the appropriate dilution prior to GC/MS analysis?	____	____
E.	Was the sample extract diluted and re-analyzed for all samples whose quantitation ion abundance exceeded the clibration range?	____	____
F.	Was the relative retention times of each detected compound within 0.06 relative retention time units of its corresponding compound in the initial calibration or calibration verification?	____	____

VI. Quality Control Parameters

		Yes	No
A.	Was an initial proficiency demonstration performed and is the data available for review?	____	____
B.	Has method detection limit (MDL) data been developed for each matrix type?	____	____
C.	Was a method blank prepared with each sample preparation batch of no more than 20 environmental samples of a specific matrix?	____	____
D.	Did the method blank contain less than the MDL of all target compounds except for the phthalate esters, which must be less than or equal to 3 times the MDL?	____	____
E.	Were blanks and QC samples processed using the same cleanup procedures as samples?	____	____
F.	Were the following surrogate compounds spiked into all samples, blanks and QA samples at the following concentrations?		

			Yes	No
1.	2-Fluorophenol	200ug/ml	____	____
2.	Phenol-d5	200ug/ml	____	____
3.	2,4,6-Tribromophenol	200ug/ml	____	____
4.	Nitrobenzene-d5	100ug/ml	____	____
5.	2-Fluorobiphenyl	100ug/ml	____	____
6.	Terphenyl-d14	100ug/ml	____	____
G.	Were the surrogate recoveries within laboratory control limits?		____	____
H.	Were samples that did not meet surrogate accuracy criteria re-analyzed and/or re-extracted and re-analyzed as required?		____	____
I.	Was a laboratory control sample (LCS) and a matrix spike/matrix spike duplicate (MS/MSD) pair prepared for each group of no more than 20 environmental samples of a specific matrix extracted within a 28 day period?		____	____
J.	For water and low level soils, did the spiking solution contain the following compounds at the following concentrations?		____	____
1.	Phenol	200 ppm	____	____
2.	2-Chlorophenol	200 ppm	____	____
3.	1,4-Dichlorobenzene	100 ppm	____	____
4.	N-Nitroso-di-n-propylamine	100 ppm	____	____
5.	1,2,4-Trichlorobenzene	100 ppm	____	____
6.	4-Chloro-3-methylphenol	200 ppm	____	____
7.	Acenaphthene	100 ppm	____	____
8.	4-Nitrophenol	200 ppm	____	____
9.	2,4-Dinitrotoluene	100 ppm	____	____
10.	Pentachlorophenol	200 ppm	____	____
11.	Pyrene	100 ppm	____	____
K.	For wastes samples, did the spiking solution contain the following compounds at the following concentrations?		____	____
1.	Phenol	1000 ppm	____	____
2.	2-Chlorophenol	1000 ppm	____	____
3.	1,4-Dichlorobenzene	500 ppm	____	____
4.	N-Nitroso-di-n-propylamine	500 ppm	____	____
5.	1,2,4-Trichlorobenzene	500 ppm	____	____
6.	4-Chloro-3-methylphenol	1000 ppm	____	____
7.	Acenaphthene	500 ppm	____	____
8.	4-Nitrophenol	1000 ppm	____	____
9.	2,4-Dinitrotoluene	500 ppm	____	____
10.	Pentachlorophenol	1000 ppm	____	____
11.	Pyrene	500 ppm	____	____
L.	Was MS/MSD and LCS accuracy criteria achieved?		____	____
M.	Were in-house performance limits used to determine if LCS, MS/MSD, and surrogate criteria was achieved?		____	____

Yes No

VII. Qualitative and Quantitative Identification

A. Was the reporting limit based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture? ___ ___

Attachment 3
QUALITY CONTROL CHECKLIST
SW-846, METHOD 8081A, ORGANOCHLORINE PESTICIDES*

		Yes	No
I.	Sample preservation, Storage & Holding Times.		
	A. Were soil samples extracted within 14 days of collection, was analysis performed within 40 days of extraction?	____	____
	B. Were water samples extracted within 7 days of collection, was analysis performed within 40 days of extraction?	____	____
	C. Were all extracts stored at 4°C in darkness until analyzed?	____	____
II.	Sample Preparation & Cleanup		
	A. Were reference standards for spike solutions obtained from independent lots?	____	____
	B. Were all QC samples (i.e. MD/MSD/LCS) extracted with their corresponding field sample?	____	____
	C. Were all QC samples and their corresponding field sample processed through the cleanup procedures?	____	____
	D. If commercially prepared Florisil columns are not being used, has the Florisil been standardized to its lauric acid value?	____	____
	E. Were duplicate checks of Florisil column recovery conducted every 300 cartridges or upon lot change?	____	____
	F. Was the Florisil column recovery of target analytes between 80-110% and was the Florisil column recovery of trichlorophenol $\leq$5%?	____	____
	G. Were all samples and QC samples from soil batches analyzed for Endrin Aldehyde prior to TBA Sulfite cleanup?	____	____
III.	Instrument Tuning & Initial Calibration.		
	A. Was an Endrin/DDT breakdown check standard run on each dissimilar column prior to standard and sample analysis and was the Endrin/DDT breakdown $\leq$15% on both columns?	____	____
	B. Was a single mid-point multi-component pesticides (toxaphene, chlordane) standard analyzed in the initial calibration sequence?	____	____
	C. Have retention time intervals been calculated for each target analyte on each column?	____	____
	D. Have the calculated retention time intervals of each target compound been applied to the absolute retention time of each individual compound in the mid-point standard of the initial calibration?	____	____

*** PCB analysis must be conducted using SW846, Method 8082**

Yes No

E. For initial calibration, one of three approaches may be taken in the following order of preference:

1. A linear 5 point calibration using the average calibration factor:

   a) Was an initial 5 point calibration for all single component analytes and surrogates analyzed on each dissimilar column and was the %RSD $\leq$ 20% for each analyte on each column? ____ ____

   b) If one or more compounds have %RSD > 20%, was the average %RSD for <u>all</u> analytes $\leq$20% on each column? (If so, the average calibration factor may be used provided the data user is provided with a summary of all analytes whose %RSD exceeded 20%). ____ ____

2. A linear calibration using least square regression. (This approach is to be used if the first option is not feasible because of prior knowledge of a detector's response or if an analyte's response always produces a %RSD> 20%. It results in a linear relationship that has a non-zero y-intercept).

   a) Was the origin not forced and not included in the regression? ____ ____

   c) Was the correlation coefficient, r^1 greater than or equal to 0.99? ____ ____

3. A non linear calibration. (This option may be used when a detector's response is known to be non-linear or when the first two options never meet criteria. It is NOT to be used for detectors that have previously demonstrated a linear response, to compensate for detector satuation or to avoid proper instrument maintenance). ____ ____

   a) Was the origin not forced and not included in the curve fitting algorithm? ____ ____

   b) Is the calibration curve continuous and does it have a tangent line whose slope is always positive or always negative? ____ ____

   c) Is the function no higher than a third order relationship? (i.e. $ax^3 + bx^2 + cx + d$)? ____ ____

   d) Was an additional standard run for each increasing order of the equation? (i.e. a second order function requires a 6 point calibration and a third order function requires a 7 point calibration). ____ ____

   e) Is the Coefficient of Determination (COD) of the equation $\geq$ 0.99 for each analyte? ____ ____

	Yes	No
F. Were surrogate compounds added to all standards?	____	____

IV. Calibration Verification

A. Was the Endrin/DDT breakdown verified on each column at the beginning of every analytical sequence and found to be ≤ 15% on both columns prior to sample analysis? (Injection port maintenance and recalibration is required if the breakdown fails on either column for either compound).	____	____
B. Was the calibration verified for all target compounds on both columns every 12 hours or every 20 samples, whichever occurs first? One of the two following options result in a successful calibration verification. If the calibration verification fails, all samples analyzed since the last successful calibration verification must be reanalyzed and a new initial calibration must be performed).	____	____
1. For a linear initial calibration using average calibration factors, calculate the % Difference (%D) of the calibration verification. Do all analytes have %D ≤ +/- 15 %? (For non-linear or linear calibrations with non-zero y-intercepts, use % Drift of the calibration verification as the parameter).	____	____
2. If all analytes %D (or %Drift) were not ≤ +/- 15% was the average of all %D's (or % Drift's) ≤ 15%? (If this option is chosen, all analytes in the calibration mix must be included and the data user must be provided with a summary of all analytes whose %D > 15%.	____	____
C. Were the average calibration factors from the original five point calibration used for quantitative analysis?	____	____
D. Were the retention times of all analytes within the retention windows established during initial calibration as required?	____	____
E. Were the retention time windows of each target analyte updated for any retention time shifts in the calibration verification standard?	____	____

V. Sample Analysis.

A. Were all samples analyzed on two dissimilar columns and were all tentative identifications confirmed on a second column?	____	____
B. If Endrin Aldehyde is a targeted compound, was it analyzed on each column prior to TBA sulfite cleanup?	____	____
C. If an interference was present in the analysis, was the appropriate extract cleanup procedure used?	____	____
D. Were the following surrogate compounds added to all samples, Blanks and QA samples at the concentrations indicated?		

		Yes	No
1. Tetrachloro-m-xylene -	50ul @ 10 ppm	____	____
2. Decachlorobiphenyl -	50ul @ 10 ppm	____	____

VI. Quality Control Parameters

	Yes	No
A. Was an Initial Demonstration of Capability (IDOC) performed and is the data available for review?	____	____
B. Has method detection limit (MDL) data been developed for each matrix type?	____	____
C. Was a method blank prepared with each sample preparation batch of no more than 20 environmental samples of a specific matrix?	____	____
D. Did the method blank contain less than ½ the quantitation limit of all target compounds?	____	____
E. Were the surrogate recoveries within laboratory control limits?	____	____
F. Were samples that did not meet surrogate accuracy criteria re-analyzed and/or re-extracted and re-analyzed as required?	____	____
G. Was a laboratory control sample (LCS) and a matrix spike/ matrix spike duplicate (MS/MSD) pair prepared for each group of no more than 20 environmental samples of a specific matrix extracted within a 28 day period?	____	____
H. Were the following compounds spiked into the LCS and MS/MSD at the indicated concentrations?		

For TCL-Pest

		Yes	No
1. Aldrin	20 ppm	____	____
2. Alpha-BHC	20 ppm	____	____
3. Beta-BHC	20 ppm	____	____
4. Delta-BHC	20 ppm	____	____
5. Gamma-BHC	20 ppm	____	____
6. 4,4'-DDD	20 ppm	____	____
7. 4,4'-DDE	20 ppm	____	____
8. 4,4'-DDT	20 ppm	____	____
9. Dieldrin	20 ppm	____	____
10. Endosulfan I	20 ppm	____	____
11. Endosulfan II	20 ppm	____	____
12. Endosulfan Sulfate	20 ppm	____	____
13. Endrin	20 ppm	____	____
14. Endrin Aldehyde	20 ppm	____	____
15. Endrin Ketone	20 ppm	____	____
16. Heptachlor	20 ppm	____	____
17. Heptachlor Epoxide	20 ppm	____	____
18. Methoxychlor	20 ppm	____	____

			Yes	No
	For TCLP/Pest			
1.	Gamma-BHC (Lindane)	5 ppm	____	____
2.	Chlordane	25 ppm	____	____
3.	Endrin	5 ppm	____	____
4.	Heptachlor	5 ppm	____	____
5.	Heptachlor epoxide	5 ppm	____	____
6.	Methoxychlor	5 ppm	____	____
7.	Toxaphene	50 ppm	____	____

I. Was MS/MSD and LCS accuracy criteria achieved? ____ ____

J. Were blanks and QC samples processed using the same cleanup procedures as samples? ____ ____

K. Are in-house performance limits used for determining if LCS, MS/MSD, and surrogate accuracy criteria has been achieved? ____ ____

VII. Qualitative & Quantitative Identification

A. Are the chromatograms adequately resolved, not overloaded and free of carryover? ____ ____

B. Were RT identification criteria met on both the primary and confirmation columns? ____ ____

C. Were all detected analytes within the calibration range of the instrument? ____ ____

VIII. Data Reporting Parameters

A. Was the reporting limit based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture? (No analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range). ____ ____

B. If the quantitative values from each column did not agree within 40%, was the discrepancy noted in the report? ("p" if it's > 40%, "*" if it's an obvious interference, and the lower of the two is reported). ____ ____

C. Was the higher of the two concentrations from each column reported for confirmed analytes? ____ ____

D. Were only Pesticide results (no PCB's) reported using method 8081A? ____ ____

Attachment 3
QUALITY CONTROL CHECKLIST
SW-846, METHOD 8082A, POLYCHLORINATED BIPHENYLS ONLY*

		Yes	No
I.	Sample preservation, Storage & Holding Times.		
	A. Were soil samples extracted within 14 days of collection, was analysis performed within 40 days of extraction?	____	____
	B. Were water samples extracted within 7 days of collection, was analysis performed within 40 days of extraction?	____	____
	C. Were all extracts stored at 4°C in darkness until analyzed?	____	____
II.	Sample Preparation & Cleanup		
	A. Were reference standards for spike solutions obtained from independent lots?	____	____
	B. Were all QC samples (i.e. MD/MSD/LCS) extracted with their corresponding field sample?	____	____
	C. Were all QC samples and their corresponding field sample processed through the same cleanup procedures?	____	____
III.	Instrument Tuning & Initial Calibration.		
	A. Were all standards analyzed on two dissimilar columns?	____	____
	B. Was an initial 5 point calibration of PCB 1221, 1232, 1242, 1248, 1254, 1262 & 1268 performed on each column?	____	____
	C. Have retention time intervals been calculated for five characteristic peaks in each Aroclor of the 1016/1260 mixture on each column?	____	____
	D. Have the calculated retention time intervals of the selected peaks been applied to the absolute retention time of each individual compound in the mid-point standard of the initial calibration?	____	____
	E. Were calibration factors generated for a minimum of five characteristic peaks in each Aroclor of the 1016/1260 mixture that have a peak height at least 25% of the largest peak in that Aroclor?	____	____
	F. For initial calibration, was a linear 5 point calibration factor using the average calibration factor applied?	____	____
	G. Was a minimum of 5 point calibration of five characteristic peaks in each Aroclor of the 1016/1260 mixture analyzed on each dissimilar column, and was the % RSD $\leq$ 20% for each analyte on each column?	____	____

*** Pesticides analysis must be conducted using SW846, Method 8081A**

Yes No

IV. Calibration Verification

A. Was the calibration verified for all selected peaks (Aroclor 1016/1260) on each column every 12 hours or every 20 samples, whichever occurs first? One of the two following options result in a successful calibration verification. If the calibration verification fails, all samples analyzed since the last successful calibration verification must be reanalyzed and a new initial calibration must be performed). ____ ____

B. Were the average calibration factors from the original five point calibration used for quantitative analysis? ____ ____

C. Were the retention times of all analytes within the retention windows established during initial calibration as required? ____ ____

D. Were the retention time windows of each target analyte updated for any retention time shifts in the calibration verification standard? ____ ____

V. Sample Analysis.

A. Were all samples analyzed on two dissimilar columns and were all tentative identifications confirmed on a second column? ____ ____

B. If an interference was present in the analysis, was an appropriate extract cleanup procedure used? ____ ____

C. Were the following surrogate compounds added to all samples, Blanks and QA samples at the concentrations indicated?

1. Decachlorobiphenyl - 50ul @ 10 ppm ____ ____

VI. Quality Control Parameters

A. Was an Initial Demonstration of Capability (IDOC) performed and is the data available for review? ____ ____

B. Has method detection limit (MDL) data been developed for each matrix type? ____ ____

C. Was a method blank prepared with each sample preparation batch of no more than 20 environmental samples of a specific matrix? ____ ____

D. Did the method blank contain less than ½ the quantitation limit of all target compounds? ____ ____

E. Were the surrogate recoveries within laboratory control limits? ____ ____

F. Were samples that did not meet surrogate accuracy criteria re-analyzed and/or re-extracted and re-analyzed as required? ____ ____

G. Was a laboratory control sample (LCS) and a matrix spike/ matrix spike duplicate (MS/MSD) pair prepared for each group of no more than 20 environmental samples of a specific matrix

	Yes	No
extracted within a 28 day period?	____	____
H. Were the following compounds spiked into the LCS and MS/MSD at the indicated concentrations?		
1. Aroclor 1016 100 ppm	____	____
2. Aroclor 1260 100 ppm	____	____
I. Was MS/MSD and LCS accuracy criteria achieved?	____	____
J. Were blanks and QC samples processed using the same cleanup procedures as samples?	____	____
K. Are in-house performance limits used for determining if LCS, MS/MSD, and surrogate accuracy criteria has been achieved?	____	____

VII. Qualitative & Quantitative Identification

A. Are the chromatograms adequately resolved, not overloaded and free of carryover?	____	____
B. Were RT identification criteria met on both the primary and confirmation columns?	____	____
C. Were all detected analytes within the calibration range of the instrument?	____	____
D. Was the average concentration of at least three PCB peaks calculated and used to determine the concentration for the reported Aroclor?	____	____

VIII. Data Reporting Parameters

A. Was the reporting limit based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture? (No analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).	____	____
B. If the quantitative values from each column did not agree within 40%, was the discrepancy noted in the report? ("p" if it's > 40%, "*" if it's an obvious interference, and the lower of the two is reported).	____	____
C. Was the higher of the two concentrations from each column reported for confirmed analytes?	____	____
D. Were only PCB results (no Pesticides) reported using method 8082?	____	____

Attachment 4
QUALITY CONTROL CHECKLIST
SW-846, METHOD 6010B, Metals by ICP

	Yes	No
1. Were all samples digested and analyzed within 180 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Was the instrument calibrated with a minimum of 3 standards?	____	____
4. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
5. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
6. Was the high standard analyzed at the beginning of the sample analysis run and were the results $\pm$5% of the true value?	____	____
7. Were Interference Check Samples (ICSA and ICSAB) run at the beginning and end of sample analysis run?	____	____
8. Was a Continuing Calibration Verification run after a maximum of 10 samples?	____	____
9. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
10. Did the Initial Calibration Verification/Continuing Calibration Verification meet the 10% control limit?	____	____
11. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the detection limit?	____	____
12. Were the results of the ICSAB within 20% of the true value?	____	____
13. Was each sample within the calibration range?	____	____
14. Did the Prep Blank meet criteria i.e. were the results less than the absolute value detection limit?	____	____
15. If the Prep Blank did not meet criteria, was the entire prep batch re-digested and reanalyzed?	____	____

	Yes	No
16. Was MS recovery within 75-125% limit?	____	____
17. Were LCS/LCSD RPD less than 20%?	____	____
18. Was a Post Analysis Spike run to demonstrate the absence of interference?	____	____
19. Was the Laboratory Control Sample within QC limits?	____	____
20. Were Sample/Duplicate RPD less than 20%?	____	____
21. Was a Serial Dilution run for each batch of samples to show the absence of interferences?	____	____
22. Were the results of the Serial Dilution within 10% of the original determination?	____	____

Attachment 5
QUALITY CONTROL CHECKLIST
SW-846, 7000 METHODS, Metals by Graphite Furnace AA

	Yes	No
1. Were all samples digested and analyzed within 180 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Were calibration standards and calibration check standards prepared daily?	____	____
4. Was the instrument calibrated with a minimum of 5 standards?	____	____
5. Was each calibration standard injected in duplicate?	____	____
6. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
7. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
8. Was a Continuing Calibration Verification run after each 10 samples?	____	____
9. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
10. Did the Initial Calibration Verification meet the 10% control limit?	____	____
10.a. Did the Continuing Calibration Verification meet the 20% control limit?	____	____
11. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the reporting limit?	____	____
12. Was each sample digestate injected in duplicate?	____	____
13. Was the duplicate injection less than 20% RPD?	____	____
14. Was each sample within the calibration range?	____	____
15. Was each sample post spiked?	____	____
16. Was the post spiked recovery within 85-115%?	____	____

	Yes	No
17. If the post spiked recovery was not within acceptable limits, was the sample run by Method of Standard Addition?	____	____
18. Were Method of Standard Addition requirements met?	____	____
19. Did the Prep Blank meet criteria i.e. were results less than the absolute value of the reporting limit?	____	____
20. If the Prep Blank did not meet criteria, was the entire prep batch re-digested and re-analyzed?	____	____
21. Was MS recovery within 75-125% limit?	____	____
22. Were LCS/LCSD RPD less than 20% for soil samples?	____	____
23. Was the Laboratory Control Sample within QC limits?	____	____
24. Were Sample/Duplicate RPD less than 20%?	____	____

Attachment 6
QUALITY CONTROL CHECKLIST
SW-846, METHOD 7471A, Mercury by Cold Vapor AA

	Yes	No
1. Were all samples digested and analyzed within 28 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Was the instrument calibrated with a minimum of 4 standards?	____	____
4. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
5. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
6. Was a Continuing Calibration Verification run after a maximum of 10 samples?	____	____
7. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
8. Did the Initial Calibration Verification meet the 10% control limit?	____	____
8.a. Did the Continuing Calibration Verification meet the 20% control limit?	____	____
9. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the reporting limit?	____	____
10. Was each sample within the calibration range?	____	____
11. Did the Prep Blank meet criteria i.e. were the results less than the absolute value of the reporting limit?	____	____
12. If the Prep Blank did not meet criteria, was the entire prep batch re-digested and re-analyzed?	____	____
13. Was MS recovery within 75-125% limit?	____	____
14. Were LCS/LCSD RPD less than 20%?	____	____
15. Was the Laboratory Control Sample within QC limits?	____	____

	Yes	No
16. Was Sample/Duplicate RPD less than 20%?	____	____

Attachment 7
QUALITY CONTROL CHECKLIST
General Chemistry

	Yes	No
1. Were all samples extracted and analyzed within the appropriate holding times?	____	____
2. Were acceptable calibration standards run?	____	____
2a. Was the correlation coefficient of the calibration within the required limit?	____	____
3. Did the method blank contain less than the detection limit of the target analyte?	____	____
4. Were all required laboratory quality control samples analyzed and the recovery within the required control limits?	____	____
5. If applicable, was an MS/MSD or Sample/Sample Duplicate analyzed and was the recovery/RPD within laboratory limits?	____	____
6. If applicable, were all environmental samples prepared within 14 days of their related MS/MSD or Sample/Sample Duplicate?	____	____
7. Were all analyte concentrations within linear range of the instrument being used?	____	____
8. Have all calculations involving dilutions been spot checked?	____	____

Attachment 8
QUALITY CONTROL CHECKLIST
USEPA SOW, VOLATILE ORGANIC COMPOUNDS BY GC/MS

	Yes	No
1. For soil or water samples preserved with HCl upon sample collection, was analysis conducted within 10 days of verified sample receipt and was the pH of the water samples recorded in the run log?	____	____
2. Did every analytical sequence commence with an injection of 50 ng of BFB that met the method specified criteria?	____	____
3. Was a 5 point initial calibration run at standard concentrations of 10, 20, 50, 100 and 200 ppb and did this calibration range meet the method specified criteria for Minimum RF and Maximum % RSD and were System Monitoring Compounds added to the standards at these concentrations?	____	____
4. If a continuing calibration check standard was run, was it run at the midpoint of the initial calibration range, did it meet the method specified criteria for Minimum RF and Maximum %D and were all subsequent samples quantitated using the RFs generated by the continuing calibration check?	____	____
5. Did the method blank contain less than the CRQL for all target compounds, except for the Methylene Chloride and Cyclohexane which must be less than 2.5 times the CRQL, and 2-Butanone and Acetone, which must be less than or equal to 5 times the CRQL?	____	____
6. Were the standard or standards run immediately after the BFB?	____	____
7. Was the method blank run immediately after the standard or standards?	____	____
8. Were all subsequent samples injected within 12 hours of the BFB injection time?	____	____
9. Were the following Internal Standards used at the following concentrations for all standards, samples, blanks and QA samples?		
1. Bromochloromethane 50 ppb	____	____
2. 1,4-Difluorobenzene 50 ppb	____	____
3. Chlorobenzene – d5 50 ppb	____	____
10. Were compounds quantitated against the method specified internal standard?	____	____

		Yes	No
11.	Were the Internal Standard areas within –50% to +100% the Internal Standard area of the calibration standard?	____	____
12.	Were the following System Monitoring Compounds added to all samples, blanks and QA samples at the following concentrations?		
	1. 1,2- Dichloroethane-d4 50 ppb	____	____
	2. Toluene-d8 50 ppb	____	____
	3. Bromofluorobenzene 50 ppb	____	____
13.	Were the System Monitoring Compounds within contract required limits?	____	____
14.	If either the Internal Standard areas or System Monitoring Compound recoveries outside acceptable limits, was the sample re-analyzed to confirm the matrix interference?	____	____
15.	Was at least one Matrix Spike/Matrix Spike Duplicate pair run per 20 environmental samples per matrix and were the following compound spiked at the following concentrations?		
	1. 1,1,-Dichloroethene 50 ppb	____	____
	2. Trichloroethene 50 ppb	____	____
	3. Benzene 50 ppb	____	____
	4. Toluene 50 ppb	____	____
	5. Chlorobenzene 50 ppb	____	____
16.	Are the chromatograms adequately resolved, not overloaded and free of carryover?	____	____
17.	Were RT and/or mass spectral identification criteria met?	____	____
18.	Were all detected analytes within the linear range of the instrument?	____	____
19.	Have all calculations involving dilutions been spot checked?	____	____
20.	Was the purge heated to 40°C for low level soil analyses?	____	____
21.	Was a storage/refrigerator blank stored and analyzed with the associated samples proving no storage contamination had occurred?	____	____
22.	For soil samples, if SW-846 method 5035 prep method was employed were the field cores preserved within 48 hours with Sodium Bisulfate solution or by freezing?	____	____

Attachment 9
QUALITY CONTROL CHECKLIST
USEPA SOW, SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS

	Yes	No
1. Were soil samples extracted within 10 days of verified sample receipt and was analysis performed within 40 days of sample extraction? Were water samples extracted within 5 days of verified sample receipt and was analysis performed within 40 days of sample extraction?	____	____
2. Did every analytical sequence commence with an injection of 50 ng of DFTPP that met the method specified criteria?	____	____
3. Was a 5 point initial calibration run at standard concentrations of 10, 25, 40, 60 and 80 ppm and did this calibration range meet the method specified criteria for Minimum RF and Maximum % RSD and were Surrogate Compounds added to the standards at these concentrations?	____	____
4. If a continuing calibration check standard was run, was it run at the 25 ppm concentration and did it meet the method specified criteria for Minimum RF and Maximum %D?	____	____
5. Did the method blank contain less than or equal to the CRQL of every target compound except the phthalate esters, which must be less than or equal to 5 times the CRQL? If the analysis is being done for the NJ Laboratory Services contract, the phthalate esters must be less than or equal to 3 times the CRQL?	____	____
6. Were 2 ul injected for all the standards, QA samples and environmental samples?	____	____
7. Were the standard or standards run immediately after the DFTPP?	____	____
8. Were all subsequent samples injected within 12 hours of the DFTPP injection time?	____	____
9. Were the following Internal Standards used at the following concentrations in all standards, samples, blanks and QA samples?		
1. 1,4-Dichlorobenzene-d4 20 ppm	____	____
2. Naphthalene-d8 20 ppm	____	____
3. Chrysene-d12 20 ppm	____	____
4. Acenapthene-d10 20 ppm	____	____
5. Phenanthrene-d10 20 ppm	____	____
6. Perylene-d12 20 ppm	____	____

	Yes	No
10. Were compounds quantitated against the method specified internal standard?	____	____
11. Were the Internal Standard areas within –50% to +100% the Internal Standard area of the calibration standard?	____	____
12. Were the following System Monitoring Compounds added to all samples, blanks and QA samples at the following concentrations?		
1. 2-Fluorophenol 150 ppm x 0.5 ml	____	____
2. Phenol-d5 150 ppm x 0.5 ml	____	____
3. 2-Chlorophenol-d4 150 ppm x 0.5 ml	____	____
4. Nitrobenzene-d5 150 ppm x 0.5 ml	____	____
5. 2-Fluorobiphenyl 150 ppm x 0.5 ml	____	____
6. 1,2-Dichlorobenzene-d4 150 ppm x 0.5 ml	____	____
7. Terphenyl-d14 150 ppm x 0.5 ml	____	____
8. 2,4,6-Tribromophenol 150 ppm x 0.5 ml	____	____
13. Were the Surrogate recoveries within contract required control limits?	____	____
14. If either the Internal Standard areas or Surrogate Compound recoveries was outside acceptable limits, was the sample re-analyzed to confirm the matrix interference?	____	____
15. Was at least one Matrix Spike/Matrix Spike Duplicate pair run per 20 environmental samples per matrix and were the following compound spiked at the following concentrations?		
1. Phenol 150 ppm x 0.5 ml	____	____
2. 2-Chlorophenol 150 ppm x 0.5 ml	____	____
3. N-Nitroso-di-n-propylamine 100 ppm x 0.5 ml	____	____
4. 4-Chloro-3-methylphenol 150 ppm x 0.5 ml	____	____
5. Acenaphthene 100 ppm x 0.5 ml	____	____
6. 4-Nitrophenol 150 ppm x 0.5 ml	____	____
7. 2,4-Dinitrotoluene 100 ppm x 0.5 ml	____	____
8. Pentachlorophenol 150 ppm x 0.5 ml	____	____
9. Pyrene 100 ppm x 0.5 ml	____	____
16. Were all environmental samples prepared within 7 days of their related MS/MSD?	____	____
17. Are the chromatograms adequately resolved, not overloaded and free of carryover?	____	____
18. Were RT and/or mass spectral identification criteria met?	____	____

	Yes	No
19. Were all detected analytes within the linear range of the instrument?	____	____
20. Have all calculations involving dilutions been spot checked?	____	____

Attachment 10
QUALITY CONTROL CHECKLIST
USEPA SOW, METHOD 200.7 CLP-M, Metals by ICP

	Yes	No
1. Were all samples digested and analyzed within 180 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Was the instrument calibrated with a minimum of 2 standards?	____	____
4. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
5. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
6. Was a CRI run at the beginning and end of sample analysis run?	____	____
7. Were Interference Check Samples (ICSA and ICSAB) run at the beginning and end of sample analysis run and every 20 samples?	____	____
8. Was a Continuing Calibration Verification run after a maximum of 10 samples?	____	____
9. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
10. Did the Initial Calibration Verification/Continuing Calibration Verification meet the 10% control limit?	____	____
11. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the CRDL?	____	____
12. Were the results of the ICSAB within 20% of the true value?	____	____
13. Was each sample within the calibration range?	____	____
14. Did the Prep Blank meet criteria i.e. were the results less than the absolute value of the reporting limit?	____	____
15. If the Prep Blank did not meet criteria, was the entire prep batch re-digested and re-analyzed?	____	____

	Yes	No
16. Was MS recovery within 75-125% limit?	____	____
17. If MS Recovery was outside the QC limit, was Post Spike run?	____	____
18. Was the Laboratory Control Sample within QC limits?	____	____
19. Was Sample/Duplicate RPD less than 20%?	____	____
20. Was a Serial Dilution run for each batch of samples?	____	____
21. Were the results of the Serial Dilution within 10% of the original determination?	____	____

Attachment 11
QUALITY CONTROL CHECKLIST
USEPA SOW, 200-M series METHODS CLP-M, Metals by Graphite Furnace

	Yes	No
1. Were all samples digested and analyzed within 180 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Were calibration standards and calibration check standards prepared daily?	____	____
4. Was the instrument calibrated with a minimum of 4 standards	____	____
5. Was each calibration standard injected in duplicate?	____	____
6. Was the duplicate injection less than 20% RPD?	____	____
7. Was the correlation coefficient (r^1) for the calibration curve at least 0.995?	____	____
8. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
9. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
10. Was a Contract Required Detection Limit Analysis run prior to sample analysis run?	____	____
11. Was a Continuing Calibration Verification run after a maximum of 20 injections?	____	____
12. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
13. Did the Initial Calibration Verification/Continuing Calibration Verification meet the 10% control limit?	____	____
14. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the CRDL?	____	____
15. Was each sample digestate injected in duplicate?	____	____

		Yes	No
16.	Was the duplicate injection less than 20% RPD?	____	____
17.	Was each sample within the calibration range?	____	____
18.	Was each sample post spiked?	____	____
19.	Was the post spiked recovery within 85-115%?	____	____
20.	If the post spiked recovery was not within acceptable limits, was the sample run by Method of Standard Addition?	____	____
21.	Were Method of Standard Addition requirements met?	____	____
22.	Did the Prep Blank meet criteria i.e. were results less than the absolute value of the CRDL?	____	____
23.	If the Prep Blank did not meet criteria, was the entire prep batch re-digested and re-analyzed?	____	____
24.	Was MS recovery within 75-125% limit?	____	____
25.	Was Sample/Duplicate RPD less than 20%?	____	____
26.	Was the Laboratory Control Sample within QC limits?	____	____

Attachment 12
QUALITY CONTROL CHECKLIST
USEPA SOW, 245.1 CLP-M, Mercury of Cold Vapor AA

	Yes	No
1. Were all samples digested and analyzed within 26 days of sample collection?	____	____
2. Were QC samples (e.g. Laboratory Control Sample, Prep Blank, Spike Sample and Sample Duplicate) digested with each batch of 20 environmental samples?	____	____
3. Was the instrument calibrated with a minimum of 5 standards?	____	____
4. Was an Initial Calibration Verification run prior to sample analysis run?	____	____
5. Was an Initial Calibration Blank run after each Initial Calibration Verification?	____	____
6. Was a Contract Required Detection Limit Analysis run prior to sample analysis run?	____	____
7. Was a Continuing Calibration Verification run after a maximum of 10 samples?	____	____
8. Was a Continuing Calibration Blank run after each Continuing Calibration Verification?	____	____
9. Did the Initial Calibration Verification/Continuing Calibration Verification meet the 20% control limit?	____	____
10. Was the absolute value of the Initial Calibration Blank/Continuing Calibration Blank less than the CRDL?	____	____
11. Was each sample within the calibration range?	____	____
12. Did the Prep Blank meet criteria i.e. were the results less than the absolute value of the CRDL?	____	____
13. If the Prep Blank did not meet criteria, was the entire prep batch re-digested and re-analyzed?	____	____
14. Was MS recovery within 75-125% limit?	____	____
15. Was Sample/Duplicate RPD less than 20%?	____	____

	Yes	No
16. Was the Laboratory Control Sample within QC limits?	____	____

STANDARD OPERATION PROCEDURE

FOR CLP ILM04.1, Preparation of Water Samples for Analysis by ICP and Furnace AA by ILM04.1 CLP Methods

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Approvals and Signatures

Laboratory Director:	Christopher A. Ouellette	Date: 4/6/04
Technical Director:	Michael J. Urban	Date: 4/1/04
QA Manager:	Madhuri R. Dave	Date: 4/1/04
Department Manager:	Chuangming Chen	Date: 3/31/04

1.0 SCOPE AND APPLICATION

1.1. The following method describes the procedure used to prepare water samples for the analysis of elemental content by Inductively Coupled Plasma Spectroscopy (ICP) and flame/furnace atomic absorption spectroscopy (AAS).

1.2. The preparation method is applicable to the determination of the following elements in water: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, and Zinc. Other elements can be analyzed using this preparation procedure provided that all method requirements are

met.

2.0 SUMMARY OF METHOD

2.1 For GFAA analysis the sample is digested in a mixture of nitric acid and hydrogen peroxide for two hours or until the digestate is reduced to a low volume of 20-25 ml.

2.2 For ICP analysis and Sb analysis by GFAA the sample is digested with a mixture of nitric acid and hydrochloric acid for two hours or until the digestate is reduced to a low volume of 20-25 ml.

3.0 INTERFERENCES

3.1. Whenever reflux is required, samples should never be allowed to boil or "bump" as this could result in significant loss of analyte. Samples must never be allowed to go to dryness or be baked.

3.2. See the related SOP's for further information related to interference

4.0 SAFETY

4.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

4.2. The analyst must wear a protective lab coat, safety glasses, and gloves when handling all samples, standards and solvents.

4.3. All questions pertaining to any safety procedure should be brought to the department manager or STL Edison Safety Officer.

4.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

4.5. PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in

the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.0 EQUIPMENT AND SUPPLIES

5.1 250 ml griffin beakers

5.2 70 mm watch glasses

5.3 75 mm funnels

5.4 Eppendorf Pipettors

5.5 Graduated cylinder

5.6 Specimen cups

5.7 Whatman # 41 filter paper or equivalent

5.8 Hot Plate

6.0 REAGENTS AND STANDARDS

6.1 REAGENTS

6.1.1 Inorganic Ventures Environmental Standards - CLPP-SPK-1, CLPP-SPK-4 (See Attachment 1).

6.1.2 Concentrated Distilled Nitric Acid -Trace Grade or equivalent.

6.1.3 Concentrated Distilled Hydrochloric Acid - Trace Grade or equivalent.

6.1.4 1:1 Nitric Acid (HNO_3)-Add equal parts of HNO_3 to water.

6.1.5 1:1 Hydrochloric Acid (HCl)-Add equal parts of HCl to water.

6.1.6 Reagent Grade Water - 18 megohm minimum .

6.1.7 Hydrogen Peroxide (30%).

6.2 STANDARDS

6.2.1 Stock Spike Standards

6.2.1.1 ICP Solutions:

CLPP-SPK-1 and CLPP-SPK-4 (Available from Inorganic Ventures, Lakewood, NJ.)

6.2.1.2 GFAA Solutions:

CLPP-PSPK-1 (Available from Inorganic Ventures, Lakewood, NJ.)

6.3 Working Spike Standards:

6.3.1 ICP Working Spike Solution-(CLP-PSPK)

Add the following to a 100 ml volumetric flask and bring to final volume of 100mls with 5% HNO_3:
10 mls each CLPP-SPK-1 and CLPP-SPK-4

6.3.2 ICP Laboratory Control Sample (LCSW)-

6.3.2.1 IV-7- from Inorganic Ventures

6.3.2.2 IV-19 from Inorganic Ventures

6.3.2.3 LCSW-III

6.3.2.4 Add 9 ml 10,000 ppm Sodium, 1ml each of 10,000 ppm Strontium and Tin. Dilute to final volume of 100 ml with 5 % HNO_3.

6.3.3 GFAA Working Spike Solution (GFAA-PSPK)

6.3.3.1 Add 10 mls of CLPF-PSPK-1 to a 100ml volumetric flask. Bring to final volume of 100 mls with 5% HNO_3.

6.3.4 GFAA Laboratory Control Sample (GFAA-LCSW)

Add 2.5 ml of IV-19. Dilute to final volume of 100 mls with 5% HNO_3 acid.

7.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

7.1 Aqueous samples must be preserved with 2 mls of concentrated nitric acid to a pH of <2 at the time of collection.

7.2 Maximum holding time for the analysis of all metals except mercury is 180 days from the Verified Time of Sample Receipt (VTSR) at the laboratory. Maximum holding time for mercury is 26 days from the Verified Time of Sample Receipt (VTSR) at the laboratory. Analysis of the samples must be completed during this time period in order to maintain compliance under this method.

8.0 QUALITY CONTROL

8.1 Quality control samples prepared with each set of samples digested include matrix spike, sample duplicate, preparation blank, and Laboratory Control Sample (LCS). Use the same environmental sample for the matrix spike and duplicate sample whenever possible. If insufficient sample is available, another environmental sample may be used for the duplicate sample. Samples identified as field blanks cannot be used for duplicate and spike sample analysis.

8.2 A Preparation Blank must be digested with every 20 samples or every

batch of samples, whichever results in the least number of samples. Label the Preparation Blank as PBS mm/dd/yy (this being the date the sample was prepared) and the batch number.

8.3 A Laboratory Control Sample (LCS) must be prepared with each group of samples digested. For GFAA aqueous samples, spike 1.0 ml of GFAA-LCSW. For ICP aqueous samples, spike 1ml each of IV-7, IV-19 and LCSW-III.

8.3.1 Results of the aqueous LCS must fall within 80-120% of the true value except for Sb and Ag. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

8.3.2 The LCS shall be labeled LCSW and the batch number for waters.

8.4 A Matrix Spike (S) and Sample Duplicate (D) must be prepared for up to each 20 samples digested.

8.4.1 Shake sample well and transfer 50 ml of sample to a 250 ml beaker for GFAA QC analysis and transfer 100 ml of sample to a 250 ml beaker ICP QC analysis.

8.4.2 Using an Eppendorf pipet, spike QA samples (S) with 1 ml of ICLP-PSPK solution (ICP predigestion spike) for samples being analyzed by ICP; and 0.5 ml of GFAA-SPK-1 (graphite furnace AA predigestion spike) for samples being analyzed by graphite furnace AA. The resulting elemental concentrations in the final solution of the spiked sample are listed in Table 1.

9.0 PROCEDURE

9.1 Acid digestion for the determination of total metals by furnace atomic absorption.

9.1.1 All glassware should be acid baked with a mixture of 5 ml HNO_3 and 5 ml HCl then thoroughly rinsed with deionized water prior to digestion.

9.1.2 Shake sample well and transfer 50 ml of sample to a 250 ml beaker. Transfer 50 ml of DI water to a 250 ml beaker labeled as PBW, and 50 ml of DI water to another beaker labeled as LCSW. For QA sample, transfer three 50 ml aliquots to three 250 ml beakers (labeled each as sample, D, and S). Spike 0.5

ml of GFAA-LCSW to the beaker labeled as LCSW; Spike 0.5 ml of GFAA-SPK to the beaker labeled as S. Add 0.5 ml of 1:1 nitric acid and 1 ml of 30% H_2O_2 to each beaker.

9.1.3 Cover the beaker with a watch glass and heat on a hot plate for two hours at 95°C or until the volume of the sample has been reduced to about 20 ml. Be certain not to allow the sample to boil.

9.1.4 After two hours have elapsed or the sample has reduced, remove the beaker from the heat and cool to room temperature. Filter sample to remove any insoluble material remaining.

9.1.5 Bring the sample volume to 50 ml with deionized water. The sample is now ready for analysis and the results are to be reported as "total metals".

NOTE: If antimony is to be analyzed by furnace AA, use the digestate produced for the ICP analysis for the determination.

9.2 Acid digestion for the determination of total metals by inductively coupled plasma analysis and/or flame atomic absorption.

9.2.1 All glassware should be acid baked with a mixture of 5 ml HNO_3 and 5 ml HCl then thoroughly rinsed with deionized water prior to digestion.

9.2.2 Shake sample well and transfer 100 ml of sample to a 250 ml beaker. Transfer 100 ml of DI water to a 250 ml beaker labeled as PBW, and 100 ml of DI water to another beaker labeled as LCSW. For QA sample, transfer three 100 ml aliquots to three 250 ml beakers (labeled each as sample, D, and S). Spike 1.0 ml each of IV-7, IV-19 and LCSW-III to the beaker labeled as LCSW; Spike 2.0 ml of CLP-PSPK solution to the beaker labeled as S. Add 2 ml of 1:1 HNO_3 and 10 of 1:1 HCl to each beaker.

9.2.3 Cover the beaker with a watch glass and heat on a hot plate for two hours at 95°C or until the volume of sample has been reduced to between 25 and 50 ml. Be certain not to allow the sample to boil.

9.2.4 After two hours have elapsed or the sample is reduced, remove

the beaker from the heat and cool to room temperature. Filter sample to remove any insoluble material remaining.

9.2.5 Bring the sample volume to 100 ml with deionized water. The sample is now ready for analysis and the results are to be reported as "total metals".

9.3 Documentation and Reporting

The technician preparing the samples is responsible for filling out the Sample Preparation Logbook and all Laboratory Chronicles pertaining to the samples digested.

10.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

11. WASTE MANAGEMENT AND POLLUTION PREVENTION

11.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

11.2. POLLUTION PREVENTION:

11.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice.

Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

11.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

12. DEFINITIONS

12.1 Refer to document DEFDOC-04 for definitions.

13. REFERENCES

USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis ILM04.1, Exhibit D Section III.

14. TABLE

TABLE 1

Spike amounts for Water Samples

ELEMENT	ICP SPIKE AMOUNT, ug/l	Furnace SPIKE AMOUNT ug/l
Aluminum	2000	
Antimony	500	100.0
Arsenic	2000	40.0
Barium	2000	
Beryllium	50.0	
Cadmium	50.0	5.0
Calcium	***	
Chromium	200	
Cobalt	500	
Copper	250	
Iron	1000	
Lead	500	20.0
Manganese	500	
Magnesium	***	
Nickel	500	
Potassium	***	
Selenium	2000	10.0
Silver	50.0	
Sodium	***	
Thallium	2000	50.0
Vanadium	500	
Zinc	500	

*** No spike required.

STANDARD OPERATION PROCEDURE

FOR EPA 200.7 CLP- ILMO4.1, Metals Analysis for Water , Wastewater Samples, and Soil Digestate by Method 200.7 CLP-M using Inductively Coupled Plasma Emission Spectroscopy

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Approvals and Signatures

Laboratory Director:	Christopher A. Ouellette	Date: 4/1/04
Technical Director:	Michael J. Urban	Date: 4/1/04
QA Manager:	Madhuri R. Dave	Date: 4/1/04
Department Manager:	Chuangming Chen	Date: 3/31/04

1.0 SCOPE AND APPLICATION

1.1. Method 200.7 CLP-M ILMO4.1 determines trace elements in solution using inductively coupled plasma emission spectrometry (ICPES). The method is applicable to all of the elements listed in Table 1. All matrices require some type of digestion/preparation step prior to an analysis on the instrument.

1.2. Detection limits, sensitivity, and the optimum linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Elements other than those listed in

Table 1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated.

Table 1

Element	Element
Aluminum	Magnesium
Antimony	Manganese
Arsenic	Molybdenum
Barium	Nickel
Beryllium	Potassium
Cadmium	Selenium
Calcium	Silver
Chromium	Sodium
Cobalt	Thallium
Copper	Vanadium
Iron	Zinc
Lead	

2.0 SUMMARY OF METHOD

A sample is digested and analyzed by Inductively Coupled Plasma. Each Sample Delivery Group of no more than 20 samples, a matrix spike, duplicate, blank and laboratory control sample is prepared and analyzed. The results are compared to control limits given in ILM04.1 Statement of Work.

3.0 INTERFERENCES

3.1 Most interferences are eliminated or greatly reduced during the acid digestion of the sample matrix.

3.2 Spectral interferences encountered on the instrument are corrected for by using baseline correction points and by applying background correction factors.

3.3 Background correction points are determined by scanning the area on either side of the wavelength and recording the apparent intensity from all other method analytes. Use single element solutions at or near the upper linear range of each element.

3.4 Interelement Correction Factors are determined by analyzing single

element solutions and recording the apparent analyte concentration.

3.5 All interfering elements must be analyzed at the same time as the elements of interest.

3.6 Physical interferences are effects associated with sample nebulization and transport. They are reduced or eliminated with the use of a peristaltic pump and internal standards. If these methods are insufficient to reduce the interference, they must be reduced by diluting the sample.

3.7 Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. The necessary rinse times must be estimated prior to sample analysis. Until required rinse times are determined a suggested rinse time of 60 seconds shall be used. If a memory interference is suspected the sample must be reanalyzed.

3.8 High salt concentrations can cause analyte signal suppressions and confuse interference tests. Dilute the sample if necessary.

4.0 SAFETY

4.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

4.2. The analyst must wear a protective lab coat, safety glasses, and gloves when handling all samples, standards and solvents.

4.3. All questions pertaining to any safety procedure should be brought to the department manager or STL Edison Safety Officer.

4.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

4.5. PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in

the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 Always add acid to water to prevent violent reactions.			
2 Exposure limit refers to the OSHA regulatory exposure limit.			

5.0 EQUIPMENT AND SUPPLIES

5.1 Thermo Jarrel Ash Model 61E Trace ICP with 486 microprocessor, monitor, printer and autosampler. Resolution 0.011 nm on a holographically grooved grating (2400 grooves/mm). Vacuum purged spectrophotometer with an axial plasma torch.

5.2 TJA 61E ICP with 486 microprocessor, monitor, printer and autosampler. The optics consist of a 0.75 m Rowland circle with a Paschen-Runge mount. The spectrometer is nitrogen purged with a self contained radio frequency generator.

5.3 Argon supply - 99.5% (Liquid)

5.4 Nitrogen supply - 99.5% (Liquid)

5.5 Operating conditions must be established by the analyst according to the instrument manufacturer's specification and must meet conditions satisfying the analytical and quality assurance requirements. These operating conditions must be archived in a hard copy form for later

reference.

6.0 REAGENTS AND STANDARDS

6.1 REAGENTS

6.1.1 18 megohm Reagent grade Type II water

6.1.2 Concentrated distilled nitric acid--Trace Grade or Equivalent

6.1.3 Concentrated distilled hydrochloric acid--Trace Grade or Equivalent

6.2 STANDARDS

6.3 Stock Solutions:

6.3.1 Calibration Standards – CLPP-CAL-1, CLPP-CAL-2, CLPP-CAL-3, and STLNJ-CAL-3 (Available from Inorganic Ventures, Lakewood, NJ.)

6.3.2 Calibration Verification Standards- QCP-CICV-1, QCP-CICV-2, QCP-CICV-3,and STLNJ-QC-3 (Available from Inorganic Ventures Inc., Lakewood, NJ)

6.3.3 Interference Check Standards – CLPP-ICS-A, CLPP-ICS-B4 (Available from Inorganic Ventures Inc., Lakewood, NJ)

6.3.4 Contract Required Detection Limit Check Standards – CLPP-CRA-1, CLPP-CRA-2, and CLPP-CRA-3 (Available from Inorganic Ventures Inc., Lakewood, NJ)

6.4 Working Solutions:

6.4.1 Working Calibration Standards

Calibration Standard (CAL2): Add 2 ml each CLPP-CAL-1, and CLPP-CAL-3, 0.4 ml of CLPP-CAL-2, and 4 ml of STLNJ-CAL-3 stock standard solutions to 200 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.2 Working Initial Calibration Verification Standard (ICV):

Add 200 µl each QCP-CICV-1 and QCP-CICV-3, 40 µ l of QCP-

CICV-2, 400 µl of STLNJ-QC-3 stock standard solutions to 100 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.3 Working Continuing Calibration Verification Standard (CCV):

Add 2 ml each QCP-CICV-1 and QCP-CICV-3, 400 µ l of QCP-CICV-2, 4 ml of STLNJ-QC-3 stock standard solutions to 100 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.4 Working Interference Check Standard A (ICSA)

Add 20 ml CLPP-ICS-A stock standard solution to 200 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.5 Working Interference Check Standard AB (ICSAB)

Add 20 ml of CLPP-ICS-A, and 2 ml of CLPP-ICS-B4 stock standard solutions to 200 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.6 Contract Required Detection Limit Check Standards (CRI)

6.4.6.1 Intermediate Contract Required Detection Limit Check Standards (CRI-INT): Add I ml of CLPP-CRA-1 and 2 ml each CLPP-CRA-2 and CLPP-CRA-3 stock standard solutions to 100 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

6.4.6.2 Working Contract Required Detection Limit Check Standards (CRI): Add 1 ml of CRI-INT solution to 100 ml volumetric flask with 5% nitric acid. Record preparation in the ICP CLP Standard Logbook.

See Attachment A for the final concentrations of all the working standards.

7.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

7.1 Aqueous samples must be preserved with 2 mls of concentrated nitric acid to a pH of <2 at the time of collection.

7.2 Soil samples must be stored at 4°C±2°C until the time of sample preparation.

7.3 Maximum holding time for the analysis of all metals except mercury is 180 days from the Verified Time of Sample Receipt at the laboratory. Maximum holding time for mercury is 26 days from the Verified Time of Sample Receipt at the laboratory. Analysis of the samples must be completed during this time period.

8.0 QUALITY CONTROL

8.1 Instrument Detection Limit (IDL) Determination

8.1.1 The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined quarterly or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying by 3 the average of the standard deviations obtained from the analysis of seven replicates of a standard 3-5 times the estimated IDL on three non-consecutive days. Instrument calibration should be verified in the following manner.

8.2 Instrument calibration should be verified in the following manner.

8.2.1 Initial calibration verification (ICV) is performed using an independent standard and a blank (ICB) immediately following the calibration at a concentration different from the calibration standards

8.2.1.1 The results of the Initial calibration verification (ICV) must agree within 90-110% of the true value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

8.2.1.2 The absolute value of the continuing calibration verification blank (ICB) must not exceed the contract required detection limit. If not, terminate the analysis, correct the problem and recalibrate the instrument.

8.2.2 Continuing calibration verification (CCV) is performed after every 10 analytical samples and at the end of the run using a standard and a blank (CCB). The standard must be at or near the midpoint of the calibration curve and at a different concentration than that used for the initial calibration verification.

8.2.2.1 The results of the check standard must agree within 90-110% of the true value. If not, terminate the analysis, correct the problem and recalibrate the instrument. Reanalyze the last 10 samples.

8.2.2.2 The absolute value of the continuing calibration verification blank (CCB) must not exceed the contract required detection limit. If not, terminate the analysis, correct the problem, recalibrate and reanalyze the last 10 samples.

8.2.3 A CRDL standard (CRI) must be run every 20 analytical samples and at the beginning and end of each run, whichever is more frequent. It must be immediately followed by the ICS analysis. The standard must be prepared at 2 times the CRDL or the IDL whichever is greater. The following analytes do not require a CRI standard to be analyzed: Al, Ba, Ca, Fe, Mg, Na, and K.

8.2.4 The interelement background correction factors are verified at the beginning and end of each run, or every 20 analytical samples, whichever is more frequent. The ICS analysis shall be immediately followed by the analysis of a CCV/CCB pair. This is done by analyzing the interference solutions.

8.2.4.1 Analytical results for analytes with CRDLs <10 ppb shall fall within ± CRDL of the analyte true value or ± IDL when the IDL is greater than the CRLD (the true value shall be zero unless otherwise stated) in the ICSA solution.

8.2.4.2 Results for the ICP analyses of ICSAB solution shall fall within 80-120% of the true value for the analytes in the solution.

8.3 One laboratory preparation blank will be analyzed with each group of samples prepared at the same time or each SDG of 20 samples, which ever is more frequent. It is conceivable to have more than one preparation blank per SDG. Results must be less than or equal to the absolute value of the CRDL.

8.3.1 If any analyte concentration in the blank is above the CRDL, the lowest concentration of that analyte in the associated samples must be 10 times the blank concentration. Otherwise, the

batch must be prepared again for the element in question and the samples reanalyzed.

8.3.2 If the concentration of the blank is below the negative CRDL, then all samples whose values are less than 10 times the CRDL must be redigested and reanalyzed.

8.4 A matrix spike is prepared and analyzed for each SDG of samples. The sample is spiked at levels indicated in Table 3, Exhibit E of ILM04.1. If analytes traditionally analyzed by furnace are analyzed by ICP the furnace spiking levels must be used (e.g. arsenic, lead, selenium, and thallium). The percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

Where SSR = Spiked sample result
SR = Sample result
SA = Spike amount

A recovery of 75-125% is required. An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits, then a post digestion spike is required to be analyzed (exception: Ag). Spike the unspiked aliquot of the sample at a concentration 2 times the indigenous level or 2 times the CRDL, whichever is greater

8.5 A duplicate is analyzed for each SDG of samples. The relative percent difference between the two results for the sample and the duplicate are determined by the following equation:

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} \times 100$$

Where RPD = Relative Percent Difference
S = Original sample result
D = Duplicate sample result

The control limit is 20% RPD for values greater than or equal to 5X the CRDL. For values < 5X CRDL the control limit is ± CRDL.

8.6 A five fold serial dilution must be performed on one sample per SDG. If the sample should contain analytes at a sufficiently high concentration; minimally a factor of 50 times above the instrumental detection limit, the results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

8.7 A Laboratory Control Sample (LCS) must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. Results of the aqueous LCS must fall within 80-120 % of the true value except for Sb and Ag. The results of the solid LCS must fall within the certified limits for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Profile and calibrate the instrument according to the instrument manufacturer's instructions using a high standard and a blank.

9.2 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated using a high standard and a blank.

10.0 PROCEDURE

10.1 Instrument Operating Parameters:

10.1.1 Set up the instrument with the operating parameters recommended by the manufacturer and as specified in the instrument operation SOPs.

10.1.2 Optimize the plasma operating conditions. This must only be done when the instrument is initially set up or when there is change in the instrument operating conditions. Follow instrument manufacturer instructions.

10.1.2.1 For the TJA 61E Trace ICP's use the following procedure:

10.1.2.2 After profiling using a 5 ppm arsenic standard. If the intensity is too low and/or the baseline is too high,

optimize the nebulizer by adjusting the nebulizer position and changing the nebulizer pressure.

10.1.2.2.1 After optimizing the nebulizer align the torch by adjusting the adjustable screws, which hold the optical path.

10.2 Instrument Performance Criteria:

10.2.1 Prior to the analysis of any samples the following must be performed.

10.2.1.1 Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference and should reflect the same change in background.

10.2.1.1.1 Background correction points must be determined during the initial set-up of the instrument. Refer to the specific instrument manual for instructions.

10.2.1.2 Interelement Correction factors must be determined annually. Refer to the instrument manufacturer recommended procedures and instrument operation SOP for instructions. Criteria for determining IEC's is an apparent positive or negative concentration for the analyte that falls within one reporting limit from zero.

10.2.1.3 The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined quarterly or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying by 3 the average of the standard deviations obtained from the analysis of seven replicates of a reagent blank signal.

10.2.1.4 A linear range verification standard must be run quarterly. The standard must be analyzed during a

routine analytical run and recovered within 5% of the true value.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data. Flush the instrument between standards and sample using the calibration blank.

10.3.2 A minimum of two exposures for each standard, sample and blank is required. The average of the exposures is reported.

10.3.3 Any analyte exceeding the linear range must be diluted and reanalyzed.

10.3.4 All interfering elements must be analyzed at the same time as the elements of interest.

10.3.5 The following analytical run sequence must be used for samples run under the ILM04.1 protocol:

Instrument Calibration(Blank and one standard)
ICV
ICB
CRI
ICSA
ICSAB
CCV
CCB
10 Samples
CCV
CCB
7 Samples
CRI
ICSA
ICSAB
CCV
CCB
Repeat until run is complete
CCV
CCB

CRI
ICSA
ICSAB
CCV
CCB

10.4 Full method required quality assurance data must be performed for each wavelength used.

10.5 All sample results must fall within the linear range. Dilute and reanalyze all samples for which the required analytes exceed the linear range as well as samples, which contain high concentrations of an interfering element.

10.6 Data Processing

10.6.1 Standard preparations must be documented in the Standard Preparation Logbook located in the metals analysis room.

10.6.2 See Operation SOP for documentation and reporting of data.

11.0 CALCULATIONS

11.1 Final Results for aqueous samples are as follows:

$$\text{Result (ug/L)} = \frac{A \times V1 \times D}{V2}$$

Where: A = Element concentration from instrument
D = Dilution performed on sample
V1 = Final volume of sample digested (in liters)
V2 = Initial volume of sample digested (in liters)

11.2 Final results for soil samples are as follows:

$$\text{Result (mg/Kg)} = \frac{A \times V1 \times D}{I}$$

Where: A = Element concentration from instrument (ppb)

D = Dilution performed on sample
V1 = Final volume of sample digested (in liters)
I = Initial dry weight of sample digested (in gram)

12.0 CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

13.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

13.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

13.2. POLLUTION PREVENTION:

13.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2.2. The quantity of chemical purchased should be based on expected

usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.0 DEFINITION

14.1 Refer to document DEFDOC-04 for definitions.

15.0 REFERENCES

15.1 USEPA Contract Laboratory Program Statement of Work for Inorganics Analysis ILM04.1, 200 Series Methods CLP-M.

16.0 TABLES

ATTACHMENT A

Working Standard Concentration for ICP Elements in ppb

Element	CAL2	ICV	CCV	CRI	ICSA	ICSAB
Aluminum	20000	2000	10000	N/A	500000	500000
Antimony	2000	200	1000	120	N/A	600
Arsenic	10000	1000	5000	20	N/A	100
Barium	20000	2000	10000	N/A	N/A	500
Beryllium	500	50	250	10	N/A	500
Cadmium	5000	500	2500	10	N/A	1000
Calcium	50000	5000	25000	N/A	500000	500000
Chromium	2000	200	1000	20	N/A	500
Cobalt	5000	500	2500	100	N/A	500
Copper	2500	250	1250	50	N/A	500
Iron	10000	1000	5000	N/A	200000	200000
Lead	10000	1000	5000	6	N/A	50
Magnesium	50000	5000	25000	N/A	500000	500000
Manganese	5000	500	2500	30	N/A	500
Nickel	5000	500	2500	80	N/A	1000
Potassium *	100000	10000	50000	N/A	N/A	N/A
Selenium	10000	1000	5000	10	N/A	50
Silver	2500	250	1250	20	N/A	200
Sodium *	250000	25000	125000	N/A	N/A	N/A
Thallium	10000	1000	5000	20	N/A	100
Vanadium	5000	500	2500	100	N/A	500
Zinc	5000	500	2500	40	N/A	1000
Boron	2000	200	1000	N/A	N/A	N/A
Molybdenum	2000	200	1000	N/A	N/A	N/A
Tin	2000	200	1000	N/A	N/A	N/A
Titanium	2000	200	1000	N/A	N/A	N/A
Strontium	2000	200	1000	N/A	N/A	N/A

* Second order curve fit is used for Sodium and Potassium. A correlation coefficient must be 0.995 or better.

METHOD NYSDEC CLP
STANDARD OPERATING PROCEDURE
FOR Mercury Analysis for Water Samples by Method 245.1 CLP-M using the Leeman Mercury Analyzer (Cold Vapor Technique)

UNCONTROLLED COPY

Approvals and Signatures

Laboratory Director:	Christopher A. Ouellette	Date: 4/1/04
Technical Director:	Michael J. Urban	Date: 4/1/04
QA Manager:	Madhuri R. Dave	Date: 4/1/04
Department Manager:	Chuangming Chen	Date: 3/31/04

1.0 SCOPE AND APPLICATION

1.1 NYSDEC CLP, Method 245.1 CLP-M is applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. This method determines total mercury, both inorganic and organic.

1.2 The typical detection limit using a 100 ml sample size is 0.2 ug/l Hg.

2.0 SUMMARY OF METHOD

2.1 A digested sample is analyzed using cold vapor atomic absorption. Each SDG of

no more than 20 samples, a matrix spike, duplicate, blank and laboratory control sample is prepared and analyzed. The results are compared to limits given in NYSDEC CLP Statement of Work.

3.0 DEFINITIONS

3.1 Refer to document DEFDOC-04 for definitions.

4.0 INTERFERENCES

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of chlorine interfering with the analysis can be minimized by using additional hydroxylamine sulfate up to 25 ml.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2. The analyst must wear a protective lab coat, safety glasses, and gloves when handling all samples, standards and solvents.

5.3. All questions pertaining to any safety procedure should be brought to the department manager or STL Edison Safety Officer.

5.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.5. PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the

MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1.000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 Mg/M3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.

Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
1 – Always add acid to water to prevent violent reactions.			
2 Exposure limit refers to the OSHA regulatory exposure limit.			

6.0. EQUIPMENT AND SUPPLIES

6.1 Glassware/Equipment:

6.1.1 300 ml BOD bottles

6.1.2 100 ml graduated cylinder

6.1.3 Eppendorf Pipettes and tips in various sizes

6.1.4 100 ml volumetric flasks

6.1.5 15 ml sample cups

6.2 Leeman Laboratories Inc. PS200 Automated Hg Analyzer

6.3 Computer and Printer with Leeman PS200 software

6.4 Analytical Balance

6.5 Pump tubing:

6.5.1 Sample, viton, blue tab

6.5.2 Reductant, red tab

6.5.3 Drain, black tab

6.6 Drying Tube - located prior to the optical cell loosely packed with >20 mesh Magnesium Perchlorate.

6.7 Nitrogen supply - capable of producing 80 PSI.

6.8 Water bath - capable of holding a temperature of 95°C.

7.0. REAGENTS AND STANDARDS

7.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent)

7.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.4 Potassium Permanganate (ACS Grade)

7.5 Potassium Persulfate (ACS Grade)

7.6 Sodium Chloride (analytical reagent grade)

7.7 Hydroxylamine Hydrochloride (ACS Grade)

7.8 Stannous Chloride (ACS Grade)

7.9 Deionized water - 18 megohm minimum

7.10 Magnesium Perchlorate, Anhydrous >20 mesh

7.11 0.5N Sulfuric acid - Cautiously add 14.0 ml of concentrated H_2SO_4 to 1 liter of deionized water.

7.12 10% Hydrochloric Acid- Cautiously add 200 mls of concentrated HCl to a container and bring to final volume of 2 liters with deionized water.

7.13 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.14 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.15 Potassium persulfate ($K_2S_2O_8$) - 5% solution w/v - Dissolve 50 g of $K_2S_2O_8$ in 1 liter of deionized water.

7.16 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.17 Stock Solutions:

7.17.1 Stock Mercury Calibration and Calibration Verification Standards –

Purchase from two different vendors.

7.18 Working Mercury Solutions:

7.18.1 Calibration Standards:

7.18.1.1 Intermediate Calibration Standard (Cal-Intermediate): Dilute 1 ml of Hg calibration stock standard (1 ml = 1 mg Hg) solution to 100 ml with deionized water = 1 mg Hg/100 ml or 10 ug Hg/ml. Record the preparation in the Mercury Standard Logbook.

7.18.1.1 Working Calibration Standard (DCAL-INT): Dilute 1.0 ml of Cal-Intermediate solution (10.4.1.1) to 100 ml with .15% HNO_3 = 10 ug Hg/100 ml = 0.1 ug Hg/ml. Record the preparation in the Mercury Standard Logbook.

7.18.2 Calibration Verification Standards:

7.18.2.1 Intermediate Calibration Verification Standard (CCV-Intermediate): Dilute 1 ml of Hg calibration stock standard (1 ml = 1 mg Hg) solution to 100 ml with deionized water = 1 mg Hg/100 ml or 10 ug Hg/ml. Record the preparation in the Mercury Standard Logbook.

7.18.2.2 Working Calibration Verification Standard (DCALV-INT): Dilute 1 ml of CCV-Intermediate solution to 100 ml with .15% HNO_3 = 10 ug Hg/100 ml = 0.1 ug Hg/ml. Record the preparation in the Mercury Standard Logbook.

7.19 Calibration Standard Preparation: Use 6 100-ml volumetric flasks to prepare the standards. Add small portion of deionized water to each flask. Working in increasing order, spike the appropriate flasks with 0.0, 0.2, 1.0, 2.0, 5.0, and 10.0 ml of working Solution DCAL-INT. Bring to final volume of 100 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively.

7.20 Mercury Initial Calibration Verification Standard Preparation: Use one 100-ml volumetric flask. Add a small portion of deionized water to the flask and spike with 3 ml of DCALV-INT. Bring to final volume of 100 ml and mix thoroughly. The corresponding concentration is 3 ppb.

7.21 Mercury Continuing Calibration Verification Standard Preparation: Use one 100-ml volumetric flask. Add a small portion of deionized water to the flask and spike with 5 ml of DCALV-INT. Bring to final volume of 100 ml and mix thoroughly.

The corresponding concentration is 5 ppb.

7.22 Mercury CRDL Standard Preparation: Use one 100-ml volumetric flask. Add a small portion of deionized water to the flask and spike with 0.2 ml of DCAL-INT. Bring to final volume of 100 ml and mix thoroughly. The corresponding concentration is 0.2 ppb.

8.0 SAMPLE COLLECTION, PRESERVATION, SHIPMENT, AND STORAGE

8.1 Soil samples are stored at 4° ± 2° C until time of analysis.

8.2. Samples are to be analyzed within 26 calendar days of verified time of sample receipt at the laboratory.

9.0 QUALITY CONTROL

9.1 Prior to the analysis of any samples the following must be performed:

9.1.1 The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined quarterly or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying by 3 the average of the standard deviations obtained from the analysis of seven replicates of a standard 3-5 times the estimated IDL on three non-consecutive days.

9.2 Instrument Calibration should be verified in the following manner.

9.2.1 The curve must be verified by analyzing an Initial Calibration Verification (ICV) solution at the midpoint of the calibration range but different from the calibration standards (3.0 ppb). The value obtained must not differ from the true value by more than 20%. If it does, the problem must be corrected, the instrument recalibrated and the ICV reanalyzed.

9.2.2 The validity of the calibration curve must be verified periodically during an analysis. A Continuing Calibration Verification (CCV) solution must be analyzed following every ten analytical sample analyses. Use a concentration of mercury at the midpoint of the calibration range but different from the calibration verification standard (5.0ppb). The value obtained for the CCV must not differ from the true value by more than 20%. If it does, the problem must be corrected and the previous ten samples reanalyzed following the last good calibration verification.

9.2.3 Following each calibration verification a calibration blank must be analyzed. The results of this analysis must fall below the contract required

detection limit. If it does not, a laboratory source contamination should be suspected, the problem corrected, instrument recalibrated and the previous ten samples reanalyzed following the last good calibration verification blank.

9.3 A preparation blank must be run for each SDG or each batch of samples digested whichever results in the least amount of samples. Use deionized water for the blank. The result of the preparation blank must be less than the absolute value of the CRDL (0.2 ppb). If the result is greater, the entire batch of samples digested with the preparation blank must be redigested.

9.4 A Laboratory Control Sample (LCSW) must be analyzed with each group of samples digested. Results of the LCSW must fall within 80 – 120%. If not, all samples prepared in association with the LCSW must be redigested and reanalyzed.

9.5 A matrix spike is prepared and analyzed with each SDG. A portion of sample is spiked with 0.1 ug of mercury (1 ml of DCAL-INT standard). This is equivalent to 1.0 ppb Hg if a 100 ml portion of sample is digested.

9.5.1 The percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{SSR - SR}{SA} \times 100$$

Where: SSR = Spiked sample result
SR = Sample result
SA = Spike amount
A recovery of 75-125% is required.

9.5.2 A duplicate is required for each SDG of samples. A duplicate sample analysis is performed on the sample used for the matrix spike if sufficient volume of sample exists. If this is not the case, choose another sample for duplicate analysis.

9.5.3 The relative percent difference is calculated using the following equation:

$$RPD = \frac{|S - D|}{(S + D)_2} \times 100$$

Where RPD = Relative Percent Difference
S = Original sample result
D = Duplicate sample result

9.5.4 The control limit is 20% RPD for values greater than or equal to 5X the CRDL. For values <5X CRDL the control limit is ± CRDL.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Calibrate the instrument according to the instrument manufacturer's instructions.

10.2 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated using five standards and a blank. One of the standards must be at the CRDL. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated.

11.0 PROCEDURE

11.1 Digestion:

11.1.1 For total mercury determination, follow steps 11.1.3-11.1.8 to digest the samples.

11.1.2 For dissolved mercury determination, a portion of sample is filtered through an all glass filtering apparatus containing a 0.45 micron filter before acidification with nitric acid to ph<2. This procedure is usually performed in the field.

11.1.3 Transfer 100 ml sample (DI water for PBW and LCSW), or standard, or an aliquot diluted to 100ml, to an appropriately identified 300 ml BOD bottle. For QA samples, transfer 3 aliquots of 100 ml samples to three BOD battles labeled as SAMPLE, D and S. Spike the LCSW with 0.5 µg of mercury (5 ml of daily DCALV-INT standard), and spike the BOD labeled as S with 0.1 µg of mercury (1 ml of DCAL-INT standard).

11.1.4 Add 5 ml concentrated H_2SO_4 and 2.5 ml concentrated HNO_3 mixing well after each addition.

11.1.5 Add 15 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color has disappeared, add additional $KMnO_4$ until the purple color persists for at least 15 minutes (Note amount of additional $KMnO_4$ in the sample preparation log). The same amount of $KMnO_4$ must be added to standards as is added to samples.

11.1.6 Add 8 ml potassium persulfate solution to each bottle.

11.1.7 Heat for 2 hours in a 95° C water bath. Remove from bath and cool.

11.1.8 Add 6 ml sodium chloride - hydroxylamine sulfate solution to reduce excess permanganate. Add 50 ml of deionized water. Mix well - solution should become colorless. If necessary additional sodium chloride - hydroxylamine HCl solution may be added. Wait at least 30 seconds after decolorization before analyzing.

11.2 Analysis:

11.2.1 Powering the Instrument:

11.2.1.1 Turn on Computer, Printer and Monitor.

11.2.1.2 Turn power on the PS200 Analyzer by pushing in the green button and the blue button along side to turn on the Hg lamp.

11.2.1.3 At C:\ICP> prompt, type PS and press enter. This will execute startup and the PS200 main menu will appear on the screen.

11.2.1.4 Plumbing the Reagent Lines:

11.2.1.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

11.2.1.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

11.2.1.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

11.2.1.4.4 Drain (Black) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

11.2.2 Preparation of Reagents and Drying Tube:

11.2.2.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

11.2.2.2 Pour the 10% HCl solution into the autosampler reservoir and submerge the autosampler arm.

11.2.2.3 Prepare the drying tube by plugging one end of the drying tube with quartz wool and filling with magnesium perchlorate. Plug the other end of the tube with quartz wool and connect to the gray drying tube fittings. Note: Do not pack tightly; the carrier gas with mercury vapor must flow freely through the quartz wool and magnesium perchlorate.

11.2.3 Start up of Instrument:

11.2.3.1 At the PS200 Main Menu Screen, press the Macro (F2) key on the keyboard.

11.2.3.2 Type WARMSTRT, press "enter", and turn on lamp. The system will wait for several minutes and turn on the pump and the gas. The message Warming Up will appear on the screen. This procedure will take approximately thirty minutes. Alternately turn on the instrument by stopping the overnight mode, and wait approximately thirty minutes for the instrument to be warmed up.

11.2.3.3 When the system is stable, a beep will sound and a System Ready message will appear on the screen. The PS200 is now ready for operation.

11.2.4 Setting the Optics (Performing the Aperture Test):

11.2.4.1 Select the following options in sequence from the PS200 main menu: (U)tility,dia(G)nostics. Using the arrow keys scroll down to aperture test and then press enter.

11.2.4.2 An aperture reading will now appear on the screen. If the value is outside +/- 100, the aperture must be adjusted.

11.2.4.3 Expose the aperture adjustment screws by removing the front panel of the PS200. The panel is easily detached by pushing it up and pulling out.

11.2.4.4 Back-out(unscrew) the aperture screw which is threaded in the furthest with an Allen wrench and then press enter. Look at the aperture reading again on the screen. Repeat this until the value stops changing (this indicates that neither aperture is blocking the

light path).

11.2.4.5 Turn the appropriate aperture screw in or clockwise 1/8th revolution (top aperture screw if the value is positive, bottom aperture screw if the value is negative) and press enter. **(Note: On a daily basis you will only need to use the bottom screw. The top screw should only need to be adjusted after major instrument maintenance.)**

11.2.4.6 Check the aperture reading and repeat this process until the reading is within +\- 100. Note: The ideal reading is zero.

11.2.5 Retrieving the Method/Data File:

11.2.5.1 Select the following options in sequence from the PS200 main menu, (P)rotocol and (G)et.

11.2.5.2 Type in the appropriate method and press enter.

11.2.5.3 The system will then prompt you for a folder name that is the data storage file. Type in a data storage file and press enter. A prompt will then say folder _____ does not exist. Create (Y or N)? Type Y and press enter. (Note: The data storage file name is the batch number with HG and the run number. e.g. 5700HG1.)

11.2.6 Autosampler/Rack Entry Setup:

11.2.6.1 Select the following options in sequence from the PS200 main menu, (A)utosampler and (R)ack entry. Type in an autosampler rack station name and press enter. At the prompt: rack _____ type does not exist. Create (Y or N)? Type Y and press "enter". (Note: The rack number is usually selected as the batch number.)

11.2.6.2 Press the Insert key and type one sample name for each sequence under the ID column while pressing "enter" after each entry.

11.2.6.3 After every tenth sample or after all the samples are entered, type QC in the next sequence number under the Macro column. This will execute a macro to run the continuing calibration verification standard and the continuing calibration blank. NOTE: Do not enter a sample name under the ID column

with the same sequence number as the QC macro or the sample will not be analyzed.

11.2.6.4 Each autosampler station rack has a total of forty four sequence numbers.

11.2.7 Analysis of Samples:

11.2.7.1 Select the following options in sequence, Menu(F1)and (S)etup.

11.2.7.2 Press "1" and enter the autosampler rack station name. The set-up screen for that rack will appear and a "Begin Cup:" prompt will be displayed at the bottom of the screen.

11.2.7.3 Enter the number of the first cup to be sampled and press "enter".

11.2.7.4 An "End Cup:" prompt will now be displayed at the bottom of the screen. Enter the number of the last cup to be sampled and press "enter". Be sure to include the QC macro as cup number.

11.2.7.5 Type (N) for name. The system will prompt you for the name of the first check sample "C1". Type CCB and enter.

11.2.7.6 The system will then prompt you for the second check sample "C2". Enter "ACCV" and enter.

11.2.7.7 Then enter the third check sample name under "C3". Enter ICV/CCV and enter. Then return to the main menu.

11.2.7.8 Select Data Output by typing (D) at the main menu. Type (S) to specify report. Type (O) to open the current report specifications and type (CONC.) to choose to report to print in concentration during analysis.

11.2.7.9 Pour out the Standards and Samples according the rack layout shown on the setup page.

11.2.7.9.1 Calibration Standards: S1(0.0ppb), S2(0.2ppb), S3(1.0ppb), S4(2.0ppb), S5(5.0ppb), S6(10.0ppb).

11.2.7.9.2 Calibration Verification Standards: C1(0.0ppb), C2(5.0ppb), C3(3.0 ppb).

11.2.7.9.3 Samples: Pour out the samples in their appropriate sequence space in the station rack.

11.2.7.10 Prior to starting the run check that the gas and pump are on and that there is fresh magnesium perchlorate in the drying tube. Recheck the aperture and adjust if necessary as described above.

11.2.7.11 Press the macro(F2) key , type in the method name and press enter. A macro will begin and sequentially run the calibration, calibration verification standards and the samples.

11.2.8 Short-Term shutdown:

11.2.8.1 Press the macro(F2) key, type OVERNITE, and press "enter".

11.2.8.2 Turn off power to the lamp.

11.2.8.3 In "overnight" mode, the pump and gas flow will turn on every few minutes, run for a few seconds, and then stop. This cycle exercises the tubes to avoid flat spots and fatigue, and the gas flow keeps the optical cell dry. Make certain that the drying tube has been loosely packed. If the drying tube is blocked, liquid may flow back into the optical cell; requiring disassembly and cleaning.

11.2.8.4 For long term shutdown check the PS200 Automated Mercury Analyzer.

11.2.9 The following analytical run sequence must be used for samples run under NYSDEC CLP SOW:

Instrument Calibration (Blank and 5 Standards)
AICV
ICB
ACCV1
CCB1
CRA
9 Samples
ACCV2
CCB2
10 Samples
ACCV3
CCB3

Repeat until run is complete
ACCV
CCB

12.0 CALCULATIONS

12.1 Final Results for aqueous samples are as follows:

$$\text{Result (ug/L)} = \frac{A \times V1 \times D}{V2}$$

Where: A= Element concentration from instrument
D= Dilution performed on sample
V1= Final volume of sample digested (in liters)
V2= Initial volume of sample digested (in liters)

13.0 METHOD PERFORMANCE

13.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

13.2 All MDL results are available on file.

14.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

14.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. POLLUTION PREVENTION:

14.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0 CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1 Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

16.0 REFERENCES

16.1. New York State Department of Environmental Conservation Analytical Services Protocol, Rev. 10/95.

16.2. Leeman PS200 Operating Manual.

STL-Edison Standard Operating Procedure	Revision Date: 04/28/04
Title: US EPA CLP OLM04.2A Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Revision 2
Laboratory Director: _____	SOP Number CLPVO04_2A04
Technical Director: _____	Page 1 of 41
QA Manager: _____	
Department Manager: _____	
File Location: F:\QAQC\SOPs\NELAC\NELAC SOPs 2004\100voams\CLPSOW\CLPVO04_2A04.DOC	

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1. SCOPE AND APPLICATION

1.1. The analytical method that follows has been designed for the determination of volatile organic compounds in aqueous and solid matrices by GC/MS techniques. The method is applicable to the compounds identified as the Target Compound List (TCL) listed in Table 1. Method performance criteria for each target analyte will be determined prior to sample analysis.

TABLE 1: Target Compound List

COMPOUND	CAS#	COMPOUND	CAS #
Acetone	67-64-1	Ethylbenzene	100-41-4
Benzene	71-43-2	2-Hexanone	591-78-6
Bromodichloromethane	75-27-4	Methylene chloride	75-09-2
Bromoform	75-25-2	4-Methyl-2-pentanone (MIBK)	108-10-1
Bromomethane	74-83-9	Styrene	100-42-5
2-Butanone (MEK)	78-93-3	1, 1,2,2-Tetrachloroethane	79-34-5
Carbon disulfide	75-15-0	Tetrachloroethene	127-18-4
Carbon tetrachloride	56-23-5	Toluene	108-88-3
Chlorobenzene	108-90-7	1, 1, 1 -Trichloroethane	71-55-6
Chloroethane	75-00-3	1, 1,2-Trichloroethane	79-00-5
Chloroform	67-66-3	Trichloroethene	79-01-6
Chloromethane	74-87-3	Vinyl chloride	75-01-4
Dibromochloromethane	124-48-1	o-Xylene	95-47-6
1,1-Dichloroethane	75-34-3	m-Xylene	108-38-3
1,2-Dichloroethane	107-06-2	p-Xylene	106-42-3
1,1-Dichloroethene	75-35-4	cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5	trans-1,3-Dichloropropene	10061-02-6
1,2-Dichloropropane	78-87-5	cis-1,3-Dichloropropene	10061-01-5
Dichlorodiflouromethane	75-71-8	1,2-Dibromoethane	106-93-4
Trichlorofluoromethane	75-69-4	Isopropylbenzene	98-82-8
Methyl Acetate	79-20-9	1,3-Dichlorobenzene	541-73-1
Tert-Butyl Methyl Ether	1634-04-4	1,4-Dichlorobenzene	106-46-7
Cyclohexane	110-82-7	1,2-Dichlorobenzene	95-50-1

COMPOUND	CAS#	COMPOUND	CAS #
Methylcyclohexane	108-87-2	1,2-Dibromo-3-chloropropane	96-12-8
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1,2,4-Trichlorobenzene	120-82-1

1.2 Contract required detection limits (CRQLs) for this method are 10ug/L for aqueous samples, 10ug/kg for low-level soil samples, and 1300ug/kg for medium level soils using purge and trap introduction techniques.

2. METHOD SUMMARY

2.1. This method is used to determine volatile organic compounds in aqueous samples as well as low or medium level soil samples. Different preparatory techniques are used depending on the matrix and level of contamination.

2.1.1. For aqueous samples, a 5mL aliquot of sample is poured into a gas-tight syringe. Internal standard solution and system monitoring compounds are added to each sample prior to analysis for use in quantitation and quality control.

2.1.2. For low-level soils, there are two sample preparatory techniques, one following the previous CLP protocol (OLM03.2) and the second following SW846 Method 5035. Following the first technique, a 5g sample is mixed with 5mL of reagent water just prior to analysis. The Method 5035 technique requires special sampling devices or containers and preservation in a buffer solution or by freezing to −12° C (+/-2°C) within 48 hours of the sampling time. Internal standard solution and system monitoring compounds are added to each sample prior to analysis for use in quantitation and quality control.

2.1.3. Medium level or methanol preserved soils are extracted with methanol, and an aliquot of the extract is added to reagent water and analyzed. Internal standard solution and system monitoring compounds are added to each sample prior to analysis for use in quantitation and quality control.

2.2. Purge and trap techniques are used to introduce the sample to the GC/MS system.

2.3. The aliquot of sample containing internal standard and system monitoring compounds is purged with helium in a closed sparging vessel. The volatile

compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column. The gas chromatograph is temperature programmed to separate the volatile organic compounds, which are then detected with a mass spectrometer.

3. INTERFERENCES

3.1. This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

3.2. The volatile laboratory has been moved to a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

3.3. The helium used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

3.4. A trip blank prepared from organic-free reagent water is carried through the sampling, storage and analysis of each group of samples to check for such contamination.

3.5. Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

3.6. Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

3.7. Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of an instrument blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain

any of the compounds found in the high level sample, the system can be considered contamination free.

3.8. The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

4. APPARATUS AND MATERIALS

4.1. Microsyringes. 10 ul to 1000 ul.

4.2. Syringes. 5 ml and 10 ml gas-tight.

4.3. Volumetric flasks. Class "A" glassware, 10 ml, 50 ml, and 100 ml.

4.4. VOA vials. 40-ml glass with PTFE –faced septum.

4.5. Vials. 2-ml amber glass with screw cap with Teflon-faced septa.

4.6. Bottles. 4oz. or 8oz. with Teflon cap liner.

4.7. Top loading analytical balance.

4.8. Spatula. Narrow, stainless steel.

4.9. EnCore sampling device. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

4.10. Stir bars. PTFE -coated, small enough to spin freely inside a VOA vial.

4.11. Purge and trap unit. Consists of three parts: the sample purge unit, the trap, and the concentrator.

4.11.1. Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required.

4.11.2. Purge and trap units used include the Tekmar 2016 automatic sampler/2000 concentrator, and the Archon 5100A automatic sampler/ OI Analytical 4560 concentrator.

4.11.3. The purge chambers of each unit are designed to accept a 5mL sample with a water column at least 3 cm deep. The headspace above the water has a volume less than 15 ml. The purge gas is introduced no more than 5 mm from the base of the water column. The purge gas passes through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin.

4.11.4. The VOCARB 3000 trap from Supelco is used. The trap is 25 cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0 cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

4.11.5. Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

4.11.6. The concentrator of each unit is capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

4.12. Gas chromatograph. HP 5890 equipped with temperature programming capability, and a flow controller capable of maintaining a constant flow rate throughout desorption.

4.13. GC column. 75 M long x 0.53 mm ID, J+W DB-624 capillary column with 3um film thickness.

4.14. Injection port liners. HP 18740-80200 or equivalent.

4.15. Mass Spectrometer (HP5970B/5971/5972): scanning from 35-300 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

4.16. GC/MS Interface: glass jet separator with fused silica transfer lines heated to 180°C.

4.17. Freezer. Capable of holding a temperature of −12° C (+/-2°C).

4.18. Data system.

4.18.1. HP Chemstation II for data acquisition.

4.18.2. HP UNIX based TARGET software for data processing. The TARGET software allows for searching any data file for ions of a specified mass, and plots ion abundance versus time or scan number. The software is capable of comparing sample spectra against reference library spectra for non-target compounds and flagging all manual edits performed by laboratory personnel.

4.18.3. Magnetic tape storage device. Allows for recording and storage of data for long-term off-line storage.

5. REAGENTS

5.1 Organic free reagent water. Distilled water purchased from Poland Spring, bubbled with nitrogen for at least two hours. Reagent water is defined as water in which an interferent is not observed at or above the CRQL of that parameter.

5.2 Methanol. Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker. (Cat. # 9077-02)

5.3 Sodium Bisulfate. Granular, ACS grade.

6. STANDARDS

6.1. Stock standards are purchased from Supelco Inc., Accustandard, or Protocol as certified mixes or prepared from neat.

6.2. Data packages documenting the identity and purity of raw materials as well as the certification of the final concentration are kept on file.

6.3. All standard solutions prepared are labeled with the standard name, concentration, date prepared, lot number and expiration date. This information is also recorded in the Standard Preparation logbook.

6.4. Stock standards in sealed ampules may be stored until the expiration date provided by the manufacturer. If no expiration date is provided, ampulated standards may be stored for up to two years.

6.5. Stock standards and working standards are stored separately at -10 °C to -20°C and are protected from light.

6.6. Working standards are prepared as follows:

Table 2: Standard Preparation

WORKING STANDARDS	CONC. OF MIX	STOCK ADDED	CONC. OF STOCK	VOLUME OF STOCK	TOTAL VOL. IN MEOH
CLP ISTD	25ppm	ISTD	1000ppm	250uL	10mL
SURR	100ppm	SURR	2500ppm	400uL	10mL
CLP VOA mix 1	100ppm	CLP VOA mix 1	1000ppm	1000uL	10mL
CLP OLM04 mix	100ppm	CLP OLM04	2000ppm	500uL	10mL
CLP ISTD SURR	25ppm	ISTD SURR	1000ppm 2500ppm	250uL 100uL	10mL
CLP MS	50ppm	CLP MS	2500ppm	200uL	10mL

6.7. Frequency of standard preparation

6.7.1. All working standards are prepared weekly or sooner if the standard shows signs of degradation or evaporation.

7. PRESERVATION AND HANDLING

7.1. Aqueous samples

7.1.1. Aqueous samples are collected in 40mL VOA vials and preserved to a pH <2.

7.1.2. Aqueous samples must be analyzed within 10 days of the Validated Time of Sample Receipt (VTSR).

7.1.3. Aqueous samples must be stored at 4°C and protected from light from the time of receipt until 60 days after delivery of the sample data package.

7.2. Soil samples

7.2.1. Soil samples are collected as bulk soil in 8oz. glass jars, in Encore sampling devices, in prepared closed-system purge and trap vials or as methanol preserved samples.

7.2.2. Bulk soil only requires refrigeration at 4°C (+/-2°C) until the time of analysis.

7.2.3. Soil samples collected in Encore sampling devices must be preserved in Sodium Bisulfate solution or frozen to −12° C (+/-2°C) within 48 hours of collection. Prepare the sample vials for sodium bisulfate preservation as follows:

7.2.3.1. Sample containers used are 40mL VOA vials with PTFE-faced septa.

7.2.3.2. Add 1 clean magnetic stir bar to each vial.

7.2.3.3. Add 1g of sodium bisulfate to each vial.

7.2.3.4. Add 5.0mL reagent water to each vial. The water and sodium bisulfate will form an acidic solution that will reduce or eliminate the biological activity in the sample, thereby preventing biodegradation of the volatile components. Enough sodium bisulfate solution should be present to ensure a pH <2.

7.2.3.5. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

7.2.4. If EnCores are to be delivered to the laboratory, collect approximately 5g of soil using the EnCore sampling device. Both ends of the device are sealed, so that there is no headspace for the volatiles to escape into.

7.2.5. Collect three EnCore samples for low-level preservation. Collect a fourth Encore sample for medium level preservation with methanol. Collect bulk soil in a 4oz. jar from each sampling point for dry weight determination.

7.2.6. Upon receipt at the laboratory, transfer the contents of 3 Encore devices to the prepared vials with sodium bisulfate solution within 48 hrs of sampling. For medium level preparation, see section 9.7.8.

7.2.7. If field preserving the low-level soil samples, 5g aliquots are added in the field to the closed-system vials prepared as in Section 7.2.3, tared and recorded, then delivered to the laboratory already preserved.

7.2.8. If the freezing technique is employed, collect EnCores per Section 7.2.4 and 7.2.5.

7.2.8.1. Upon receipt at the laboratory, transfer the contents of each EnCore to empty VOA vials.

7.2.8.2. Add stir bars to the vials to be used for low level analysis.

7.2.8.3. Freeze the vials at −12° C (+/-2°C) within 48 hrs of sampling.

7.2.9. Methanol preserved soils are sampled as either 5g aliquots into 10ml of prepared methanol or 10g aliquots into 25ml of prepared methanol into 40ml VOA vials.

7.2.10. Re-weigh sample vials to determine the actual sample weight. Record the sample weight on the sample vial.

7.2.11. All soil samples must be analyzed within 10 days of the Validated Time of Sample Receipt (VTSR).

7.2.12. Soil samples must be stored at 4°C and protected from light from the time of receipt until 60 days after delivery of the sample data package.

7.2.13. For Method 5035 low-level field preservation, if samples are known or suspected to contain high levels of carbonates, a test sample should be added to a vial containing sodium bisulfate solution to check for effervescence.

7.2.13.1. If effervescence is seen, collect the soil without preservative and the samples will be analyzed within 48 hrs or frozen.

7.3. Refrigerator blanks

7.3.1. The storage of all samples is monitored through the analysis of refrigerator blanks.

7.3.2. Refrigerator blanks are placed into the storage refrigerators and stored for one week. The refrigerator blanks are analyzed for background contamination. If background contamination is detected, an assessment is of samples analyzed during past week is conducted to determine the extent of contamination.

7.3.3. The storage refrigerator is taken out of service, cleaned out and re-assessed with refrigerator blanks.

7.3.4. The refrigerator is not placed back into service unless the refrigerator blank analysis demonstrates that contamination during storage is no longer occurring.

7.4. Storage blanks

7.4.1. Upon receipt of the first sample in a sample delivery group, two 40mL VOA vials are filled with reagent water. These storage blanks are kept with the samples in the SDG and stored under the same conditions. After all samples in the SDG have been analyzed, the storage blank is analyzed. The storage blank will indicate whether any contamination has occurred during storage.

8. SAFETY

8.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. The analyst should wear the appropriate personal protective equipment including lab coat, safety eyewear, and gloves.

8.3. Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

8.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

8.5 PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9. PROCEDURE

9.1. Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure.

9.1.1. Instrument operating parameters:

Purge and trap unit:

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Helium
Purge Flow:	40-45 ml/min
Purge Temp:	Water : Ambient; Solids : 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C

Gas chromatograph:

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min
Oven Program:	35° for 3 mins., 4°C/min to 75°C, 15°c/min to 250°C
Run Time:	30 Minutes

Mass Spectrometer:

Electron Energy:	70 volts (nominal)
Mass Range:	35-300amu
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

9.2. Instrument Tuning

9.2.1. The GC/MS tune is checked at the beginning of each 12 hour shift (prior to the injection of calibration standards or blanks) by injecting 1 ul of 50 ppm BFB to meet the following criteria:

TABLE 3: BFB Key Ions and Abundance Criteria

Mass	Ion Abundance Criteria
50	8.0-40.0 % of the base peak
75	30.0-66.0% of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 % of the base peak
173	Less than 2.0% of mass 174
174	50.0-120.0 % of mass 95
175	4.0-9.0% of mass 174
176	93.0-101.0% of mass 174
177	5.0-9.0 percent of mass 176

9.2.2. The spectrum for BFB must be acquired in the following manner: the peak apex and the scans immediately before and after the apex are averaged. Background subtraction is required and must be accomplished using a single scan not more than 20 scans from the beginning of the peak.

9.2.3. A successful BFB must be acquired before calibration may begin.

9.3. Initial Calibration

9.3.1. Once the BFB has been injected and met the criteria in section 9.2.1, calibrate the GC/MS system.

9.3.2. The GC/MS system is calibrated by analyzing five calibration standards containing all of the target compounds and system monitoring compounds. The calibration standards are prepared at the following concentrations: 10, 20, 50, 100, and 200ppb.

9.3.3. A unique initial calibration must be prepared for each sample introduction technique. Aqueous and medium level soil calibrations will require ambient purge temperature; low level soil calibrations require 40°C purge temperature. The calibration standards for the Method 5035 sample preparatory technique should contain the same amount of Sodium Bisulfate as is used to preserve the samples if sodium bisulfate was used as a preservative.

9.3.4. Aqueous and/or medium level soil calibrations

9.3.4.1. Prepare aqueous calibration standards at five concentration levels for each parameter to be measured by adding the following amounts of each working standard to a 5mL syringe of reagent water:

TABLE 4: INITIAL CALIBRATION PREPARATION

STANDARD	CONC.	200ppb	100ppb	50ppb	20ppb	10ppb
CLP ISTD	25ppm	10uL	10uL	NA	10uL	10uL
SURR	100ppm	10uL	5uL	NA	1uL	0.5uL
CLP mix 1	100ppm	10uL	5uL	2.5uL	1uL	0.5uL
CLP OLM04	100ppm	10uL	5uL	2.5uL	1uL	0.5uL
ISTD/SURR	25ppm	NA	NA	10uL	NA	NA
MEOH Comp.		NA	15uL	25uL	27uL	28.5uL

9.3.4.2. Compensation methanol is added to keep the total volume of methanol purged at a constant.

9.3.4.3. The total volume of methanol is not to exceed 2% by volume.

9.3.4.4. Load the aqueous calibration standard into a purge vessel and proceed to section 9.3.7.

9.3.5. Low-level soil calibration (using CLP 3/90 sample prep. or freezing preservation – SW-846 Method 5035)

9.3.5.1. Prepare the calibration standards in a 5mL syringe as in section 9.3.4.1. Add the contents of the syringe to a 40mL VOA vial containing 5g of purified solid matrix. Proceed to section 9.3.7. (Up to 10ml of reagent water may be used for low level soil analysis to increase purge gas/sample interaction)

9.3.6. Low-level soil calibration (using sodium bisulfate preservation - SW846 Method 5035)

9.3.6.1. Prepare the calibration standards in a 5mL syringe as in section 9.3.4.1. Add the contents of the syringe to a 40mL voa vial already containing 5g of purified solid matrix, 5mL of reagent water, 1g Sodium Bisulfate, and a magnetic stir bar. Proceed to section 9.3.7. (Up to 10ml of reagent water may be used for low level soil analysis to increase purge gas/sample interaction)

9.3.7. Purge the standard for 11 minutes. Aqueous and medium level calibrations are purged at ambient temperature; low-level soil calibrations are purged at 40°C.

9.3.8. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C and backflushing it with helium.

9.3.9. Begin the GC temperature program and data acquisition.

9.3.10. *Re-condition the trap by baking at 260°C for 12 minutes.*

9.3.11. Cool the trap to (<31°C). The trap is now ready for the next sample.

9.3.12. Transfer data to network, and process using TARGET software.

9.4. Evaluation of the initial calibration

9.4.1. Internal standard calibration is used for this method.

9.4.2. Each target analyte, surrogate, and internal standard is quantitated based on the integrated area of a characteristic ion or quant ion. See Table 5 for quant ions of target analytes.

Table 5: Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
Dichlorodifluoromethane	85	87
Chloromethane	50	52

Parameter	Primary ion	Secondary ion
Bromomethane	94	96
Vinyl chloride	62	64
Chloroethane	64	66
Trichlorofluoromethane	101	103
Methylene chloride	84	49,51,86
Acetone	43	58
Carbon disulfide	76	78
1,1,2-Trichloro-1,2,2-Trifluoroethane	101	85,151
Methyl Acetate	43	74
Trans-1,2-Dichloroethene	96	61,98
Tert-Butyl Methyl Ether	73	43,57
1,1-Dichloroethene	96	61,98
Cis-1,2-Dichloroethene	96	61,98
1,1-Dichloroethane	63	65,83,85,98,100
1,2-Dichloroethene	96	61,98
Chloroform	83	85
1,2-Dichloroethane	62	64,100,98
2-Butanone	43	57,72
1,1,1-Trichloroethane	97	99,117,119
Cyclohexane	56	69,84
Carbon tetrachloride	117	119,121
Bromodichloromethane	83	85
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,2-Dichloropropane	63	65,114
trans,-1,3-Dichloropropene	75	77
Trichloroethene	130	95,97,132
Methylcyclohexane	83	55,98
Dibromochloromethane	129	208,206
1,1,2-Trichloroethane	97	83,85,99,132,134
Benzene	78	--
cis-1,3-Dichloropropene	75	77
Bromoform	173	171,175,250,252
2-Hexanone	43	58,100
4-Methyl-2-Pentanone	43	58,100
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,2-Dibromoethane	107	109

Parameter	Primary ion	Secondary ion
Chlorobenzene	112	114
Ethylbenzene	106	91,
Styrene	104	78,103
Total Xylenes	106	91
Isopropylbenzene	105	120,77
1,3-Dichlorobenzene	146	111,75
1,4-Dichlorobenzene	146	111,75
1,2-Dichlorobenzene	146	111,75
1,2-Dibromo-3-chloropropane	75	157,155
1,2,4-Trichlorobenzene	180	182,145
Surrogate Standards		
4-Bromofluorobenzene	95	174,176
1,2-Dichloroethane-d4	65	102
Toluene-d8	98	70,100
Internal Standards		
Bromochloromethane	128	130
Chlorobenzene-d5	117	82,119
1,4-Difluorobenzene	114	63,88

9.4.3. In certain situations, the GC/MS software may not correctly integrate a target compound. In these cases, a manual integration must be performed. This manual integration is to include only the area of the compound above the baseline and between the points where the sides of the peak intersect the baseline.

9.4.4. In all instances of manual integration, the analyst must identify such edits by initialing and dating the report and including a printout of the extracted ion current profile showing the manual integration.

9.4.5. Calculate the relative response factor (RRF) for each compound using the equation below. This calculation is performed automatically using the Target Software package.

$$RRF = \frac{As \times Cis}{Ais \times Cs}$$

where:

As = Peak area (or height) of the analyte or surrogate.
Ais = Peak area (or height) of the internal standard.
Cs = Concentration of the analyte or surrogate.
Cis = Concentration of the internal standard.

9.4.6. Calculate the mean RRF and the relative standard deviation (RSD) of the five RRFs for each compound in the calibration curve.

9.4.7. The relative response factor at each concentration level must be greater than or equal to the compound's minimum acceptable response factor listed in Table 7.

9.4.8. The % RSD for each compound must be less than or equal to the RSD value listed in Table 7.

9.4.9. Up to two compounds may fail the criteria listed in sections 9.4.7 and 9.4.8 provided they have a minimum RRF greater than 0.0100 and the %RSD is be less than or equal to 40.0%.

TABLE 7: INITIAL AND CONTINUING CALIBRATION CRITERIA

Compound	Min.RRF	Max%RSD	Max%Diff
Dichlorodifluoromethane	0.010	none	none
Chloromethane	0.010	none	none
Bromomethane	0.100	20.5	25.0
Vinyl chloride	0.100	20.5	25.0
Chloroethane	0.010	none	none
Trichlorofluoromethane	0.010	none	none
Methylene chloride	0.010	none	none
Acetone	0.010	none	none
Carbon disulfide	0.010	none	none
Methyl Acetate	0.010	none	none
1,1-Dichloroethene	0.100	20.5	25.0
1,1,2-Trichloro-1,2,2-trifluoroethane	0.010	none	none
1,1-Dichloroethane	0.200	20.5	25.0
trans-1,2-Dichloroethene	0.010	none	none
Methyl tert-Butyl Ether	0.010	none	none

Compound	Min.RRF	Max%RSD	Max%Diff
cis-1,2-Dichloroethene	0.010	none	none
Chloroform	0.200	20.5	25.0
1,2-Dichloroethane	0.100	20.5	25.0
2-Butanone	0.010	none	none
1,1,1-Trichloroethane	0.100	20.5	25.0
Cyclohexane	0.010	none	none
Carbon tetrachloride	0.100	20.5	25.0
Bromodichloromethane	0.200	20.5	25.0
1,2-Dichloropropane	0.010	none	none
cis-1,3-Dichloropropene	0.200	20.5	25.0
Trichloroethene	0.300	20.5	25.0
Methyl Cyclohexane	0.010	none	none
Dibromochloromethane	0.100	20.5	25.0
1,2-Dibromoethane	0.010	none	none
1,1,2-Trichloroethane	0.100	20.5	25.0
Benzene	0.500	20.5	25.0
trans-1,3-Dichloropropene	0.100	20.5	25.0
Bromoform	0.100	20.5	25.0
4-Methyl-2-pentanone	0.010	none	none
2-Hexanone	0.010	none	none
Tetrachloroethene	0.200	20.5	25.0
Isopropylbenzene	0.010	none	none
1,1,2,2-Tetrachloroethane	0.300	20.5	25.0
Toluene	0.400	20.5	25.0
Chlorobenzene	0.500	20.5	25.0
Ethylbenzene	0.100	20.5	25.0
Styrene	0.300	20.5	25.0
Xylenes(total)	0.300	20.5	25.0
1,3-Dichlorobenzene	0.600	20.5	25.0
1,4-Dichlorobenzene	0.500	20.5	25.0
1,2-Dichlorobenzene	0.400	20.5	25.0
1,2-Dibromo-3-chloropropane	0.010	none	none
1,2,4-Trichlorobenzene	0.200	20.5	25.0
System Monitoring Compounds			
Bromofluorobenzene	0.200	20.5	25.0
1,2-Dichloroethane-d4	0.010	none	none
Toluene-d8	0.010	none	none

9.4.10. Initial calibration technical acceptance criteria must be met before any blanks, samples, or QA samples can be analyzed.

9.4.11. If time remains in the 12-hour period after a successful calibration, sample analysis (following a successful method blank) may take place. It is not necessary to analyze a continuing calibration standard provided that the 50ug/L standard from the initial calibration meets the technical acceptance criteria for the continuing calibration. All quantitation shall be done using the response factors from the 50ug/L standard.

9.4.12. If a successful initial calibration can not be acquired, corrective action must be taken. This may include cleaning the ion source, changing the GC column, or servicing the purge and trap unit.

9.5. Continuing calibration

9.5.1. Calibration verification must be performed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification begins with the analysis of a BFB instrument tune check followed by the analysis of a continuing calibration standard.

9.5.2. Tune Verification. Follow the procedure for the instrument tune described in section 9.2, using a 50 ng injection of BFB. If the tune cannot be verified analysis must be stopped, corrective action taken and a return to "control" demonstrated before *continuing with the calibration verification process.*

9.5.3. Calibration Verification. Analyze the continuing calibration standard (50ug/L) immediately after a BFB that meets criteria. NOTE: The same sample introduction technique employed for the initial five- point calibration must be used for the continuing calibration.

9.5.4. Calculate relative response factors (RRF) for each compound using the internal standard method as in section 9.4.5.

9.5.5. Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.5.6. The relative response factor for each compound must be greater than or equal to the minimum RRF value listed in Table 7.

9.5.7. The %Difference for each compound must be less than or equal to the maximum % D value listed in Table 7.

9.5.8. Up to two compounds may fail the requirements listed in sections 9.5.6 and 9.5.7 provided these two compounds have a minimum relative response factor greater than or equal to 0.010 and the % D is less than 40%.

9.5.9. A successful continuing calibration must be acquired before sample analysis may continue. If a successful continuing calibration cannot be acquired, corrective action must be taken. Re-calibrate the instrument as in section 9.3. The instrument may require other maintenance in order to achieve successful calibration.

9.5.10. All quantitation following the continuing calibration is done using response factors from the continuing calibration standard.

9.6. Blank analysis

9.6.1. Three different types of blanks are required by this method, the storage blank, the method blank, and the instrument blank.

9.6.1.1. A storage blank is defined as a 40mL VOA vial filled with reagent water that is stored along with the samples from a particular SDG. At least one storage blank per SDG must be analyzed after all samples for that SDG have been analyzed.

9.6.1.2. A method blank is defined as a volume of reagent water or a volume of purified solid matrix that is carried through the entire analytical procedure. The method blank must be analyzed immediately following successful calibration and before sample analysis.

9.6.1.3. An instrument blank is an aliquot of reagent water analyzed following a sample that contains a target compound that exceeds the calibration range. The

instrument blank must be analyzed in the same autosampler position as the saturated sample. The analysis of instrument blanks is repeated until the instrument blank meets the technical acceptance criteria for the method blank analysis.

9.6.2. Analyze a method blank every 12 hours of sample analysis, immediately after successful calibration.

9.6.3. Analyze the method blank in the sample manner as the associated samples will be analyzed, 5mL of reagent water for aqueous samples, 5g solid matrix for soil samples, and 4g solid matrix extracted with 10ml methanol for medium level soils.

9.6.3.1. Prepare an aqueous blank by adding 10 ul of ISTD and 2.5uL of system monitoring solution to a syringe containing 5 ml of reagent water.

9.6.3.2. Prepare a medium or high level blank by extracting 4g of a purified solid matrix with 10 mL methanol. Cap and shake for two minutes. A 100uL aliquot of the extract is added to 5mL reagent water and spiked with adding 10uL of IS and 2.5uL of system monitoring compound solution.

9.6.3.3. Prepare a low- level soil blank by weighing out 5g of clean solid matrix and adding 5mL reagent water to which 10 uL of IS and 2.5uL of system monitoring compound solution has been added.

9.6.3.4. Prepare a low- level soil blank for the Method 5035 prep by weighing out 5g of clean solid matrix into a voa vial containing 5ml reagent water, 1g Sodium Bisulfate and a stir bar. Add 5mL reagent water to which 10 uL of IS and 2.5uL of system monitoring compound solution has been added.

9.6.3.5. Up to 10ml of reagent water may be used to increase purge gas/sample interaction.

9.6.3.6. Analyze the blank as in section 9.3.7.

9.6.4. Technical acceptance criteria for blank analysis

9.6.4.1. All blanks must be analyzed on a GC/MS system meeting all requirements for BFB and calibration.

9.6.4.2. All blanks must have acceptable recoveries for the system monitoring compounds.

9.6.4.3. All blanks must meet the acceptance criteria for internal standard response and retention time as in section 10.6.

9.6.4.4. The blank must have no target compounds found above the CRQL, with exceptions made for four compounds--methylene chloride and cyclohexane are allowed to be up to 2.5 times the CRQL, and acetone and 2-butanone are allowed to be up to 5 times the CRQL.

9.6.4.5. Any method blank or instrument blank that fails to meet these criteria must be re-analyzed. Any samples or QC samples acquired within a 12-hour period in which the method blank fails criteria must be re-analyzed.

9.7. Sample analysis

9.7.1. Sample analysis may begin after successful calibration and blank analysis.

9.7.2. Equilibrate all samples to room temperature prior to analysis. Thaw frozen soil samples on the day of analysis.

9.7.3. All samples are screened prior to analysis by GC/FID static headspace analysis using Method 5021 to provide the analyst with appropriate dilution factors.

9.7.4. Aqueous samples

9.7.4.1. Carefully pour the sample into the barrel of a syringe, just short of overflowing. Replace the plunger, and compress the sample. Vent any air trapped in the syringe, and adjust the sample to volume.

9.7.4.2. Add 10uL of 25ppm IS solution and 2.5uL of 100ppm system monitoring compound solution to the syringe.

9.7.4.3. Inject the aqueous sample into the purge vessel.

9.7.4.4. Purge each sample for 11 minutes.

9.7.4.5. The volatile components of each sample are swept out of the purge chamber to the trap.

9.7.4.6. Once purging is complete, the trap is heated and backflushed with helium. The volatiles are desorbed onto the GC column. The GC is temperature programmed to separate the components, which are then detected with the mass spectrometer.

9.7.4.7. Data is transferred to the Target network for processing and evaluation.

9.7.4.8. The process of taking an aliquot of sample destroys the validity of that sample for future volatile analysis. If there is only one vial of sample, immediately fill a second syringe and hold it until it can be determined that the first analysis has been successful.

9.7.4.9. Once sample aliquots have been taken from the VOA vial, the pH of each aqueous sample must be determined to ensure that samples were properly preserved. Test the pH by placing one or two drops of sample on narrow range pH paper. Record the pH of each sample and report in the SDG narrative.

9.7.5. Low-level soil samples (bulk soil)

9.7.5.1. The sample for volatile organics is defined as the entire contents of the sample container. Do not discard any supernatant liquids. Mix the sample with a narrow spatula. Weigh out 5g of soil into a 40mL VOA vial.

9.7.5.2. If on-column concentration of any target compound exceeds the calibration range, a smaller sample size of down to 0.5g is permitted.

9.7.5.3. Spike 10mL of reagent water with 10uL of 25ppm IS and 2.5uL of 100ppm system monitoring compound solution.

9.7.5.4. Add the spiked reagent water to the 5g portion of soil immediately prior to purging.

9.7.5.5. Sample vials are placed in the autosampler tray and the unit is scheduled accordingly.

9.7.5.6. Purge each sample for 11 minutes, while heating to 40°C.

9.7.5.7. The volatile components of each sample are swept through a heated transfer line to the trap.

9.7.5.8. Once purging is complete, the trap is heated and backflushed with helium. The volatiles are desorbed onto the GC column. The GC is temperature programmed to separate the components, which are then detected with the mass spectrometer.

9.7.5.9. Data is transferred to the Target network for processing and evaluation.

9.7.5.10. After each soil sample has been analyzed, determine the % moisture. All concentrations and quantitation limits for soils will be adjusted accordingly.

9.7.5.10.1. Weigh 5-10g of soil into a tared crucible. Dry overnight in a drying oven at 105°C. Re-weigh the crucible and calculate the % moisture.

$$\%\text{moisture} = \frac{\text{g wet weight - g dry weight}}{\text{g wet weight}} \times 100$$

9.7.6. Low-level soils (Method 5035 prep – sodium bisulfate preservation)

9.7.6.1. Low-level soils collected in Encore sampling devices are preserved in Sodium Bisulfate solution within 48 hours of collection. Just prior to analysis, add 5mL of reagent water containing 10uL of IS solution and 2.5uL of system monitoring compound solution to the VOA vial containing the sample. This addition is done through the septum of the VOA vial to prevent any loss of volatile compounds in the vial.

9.7.6.2. Proceed as in section 9.7.5.5.

9.7.7. Low-level soils (Method 5035 prep – freezing preservation)

9.7.7.1. Thaw frozen soil sample.

9.7.7.2. Proceed as in section 9.7.5.3.

9.7.8. Medium level soils

9.7.8.1. The medium level soil procedure is based on extracting the soil sample with methanol. An aliquot of the methanol extract is added to reagent water containing internal standard and system monitoring compounds. The medium level sample is purged at ambient temperature and may be analyzed using the same initial and/or continuing calibration as aqueous samples.

9.7.8.2. The soil sample for volatile organics is defined as the entire contents of the sample container. Do not discard any supernatant liquids. Mix the sample with a narrow spatula.

9.7.8.3. Weigh out 4.0g of sample into a tared vial. Quickly add 10mL of methanol to the vial.

9.7.8.4. If EnCores were collected and either methanol preserved upon receipt or frozen, a 5g aliquot is used instead of

4g. The frozen aliquot may be thawed on the day of analysis and extracted in methanol.

9.7.8.5. Cap and shake for two minutes. These steps must be performed rapidly to prevent to loss of volatiles.

9.7.8.6. Allow the soil to settle, and transfer the methanol to a 2mL amber vial for storage without headspace.

9.7.8.7. Inject 100 ul of the extract or an appropriate aliquot determined from screening into a 5 ml syringe containing reagent water spiked with 10uL of 25ppm IS solution and 2.5ul of 100ppm system monitoring compound solution.

9.7.8.8. Load into the purge vessel and analyze as in section 9.7.4.4 - 9.7.4.8.

9.7.8.9. Determine the % moisture as in section 9.7.5.10.

9.7.9. Field preserved methanol soil samples

9.7.9.1 Proceed as in section 9.7.8.7 after screening to determine appropriate dilution factor.

9.7.10. Sample dilutions

9.7.10.1. If the on-column concentration for any target compound exceeds the calibration range, a dilution is required.

9.7.10.2. All dilutions for aqueous samples are made in volumetric flasks. Inject the proper aliquot from the second sample syringe into a volumetric flask containing reagent water. Aliquots of less than 1mL are prohibited. Dilute to volume, cap, and invert three times.

9.7.10.3. If a dilution is required for a low-level bulk soil, a smaller aliquot of soil can be used. Aliquots down to 0.5g are permitted.

9.7.10.4. If a dilution is required for a low-level soil prepared by the Method 5035 method, proceed to the medium level method.

9.7.10.5. If a dilution is required for a medium level soil, inject a smaller portion of the methanol extract into the 5mL syringe. Compensate with blank methanol to keep the total volume of methanol constant for all analyses.

9.7.10.6. Use the original analysis to determine the appropriate dilution factor, keeping the response of the largest target compound in the upper half of the calibration range.

9.7.10.7. Submit data from no more than two analyses.

9.7.10.8. After a sample that contained a target compound at a level exceeding the calibration range, the instrument must be proven free of carryover.

9.7.10.9. Analyze an instrument blank immediately after the saturated sample in the same autosampler position. The instrument blank must meet the technical acceptance criteria for the method blank in section 9.6.4.

9.7.10.10. Alternatively, monitor the sample analyzed immediately after the saturated sample for any of the compounds that exceeded the calibration range. This sample must not contain any of the saturated compounds above the CRQL. If an autosampler was used, the next sample to use the same position must also meet these criteria.

9.8. Data processing

9.8.1. Prior to processing any standards or samples, methods and sublists must be assembled in the Target system. These methods include compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

9.8.2. Key data is manually entered the first time a method is used for data processing. Processing data using a particular method automatically generates response factor data and updates retention time information.

9.8.3. Data is transferred from the acquisition PC to the network for processing with TARGET software.

9.8.3.1. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

9.8.3.2. Each data file is processed using response factors from the most recent continuing calibration. If samples were analyzed beneath an initial calibration, use response factors from the 50ug/L standard.

9.8.4. Target compound reporting

9.8.4.1. Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by the same method. Compounds are identified as present when the following criteria are met:

9.8.4.1.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

9.8.4.1.2. All ions present in the standard mass spectra at a relative intensity of greater than 10% must be present in the sample spectrum.

9.8.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

9.8.4.1.4. The relative intensities of the characteristic ions agree within 20% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 30% and 70%).

9.8.4.2. Quantitative analysis for target compounds is performed using the internal standard method.

9.8.4.2.1. Integrate the abundance of the primary ion for each compound.

9.8.4.2.2. Determine the concentration using the response factor for each analyte as follows:

$$\frac{As \times Cis}{Ais \times RF} = \text{conc. of compound}$$

where:

As = Peak area of the analyte or surrogate.
Ais = Peak area of the internal standard.
Cis = Concentration of the internal standard.
RF = response factor for each compound

9.8.4.3. All compounds meeting the identification criteria shall be reported with their spectra. Concentrations below the CRQL will be flagged "J".

9.8.5. Non-target compound reporting

9.8.5.1. Up to 30 compounds of the greatest apparent concentration shall be reported as non-target compounds. Do not include Carbon dioxide, semi-volatile target compounds, or any peak that elutes earlier than 30 seconds before the first target compound or 3 minutes after the last target compound. Do not report any compound that has a response less than 10% of the nearest internal standard.

9.8.5.2. Up to 20 peaks of greatest apparent concentration that are suspected to be straight-chain, branched or cyclic alkanes, alone or part of an alkane series shall be searched and reported as well. Alkanes do not count as part of the 30 organic compounds described above.

9.8.5.3. Qualitative identification of non-target compounds is done using the most recent version of the Wiley library. The following guidelines are used for identifying non-target compounds:

9.8.5.3.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

9.8.5.3.2. The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).

9.8.5.3.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

9.8.5.3.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

9.8.5.3.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

9.8.5.4. If no valid identification of the non-target compound can be made, each TIC should be reported as follows:

9.8.5.4.1. If the library search reported a match with a quality value above 85%, report that compound.

9.8.5.4.2. If the library search reported more than one compound with a quality value above 85%, report the compound with the highest quality value.

9.8.5.4.3. If the library search did not report any compound with a quality value above 85%, report the compound as "unknown". If the type or class of compound can be determined, include it in the name. If the molecular weight can be determined, include it as well.

9.8.5.5. Straight-chain, branched or cyclic alkanes are not to be reported as tentatively identified compounds with the 30 compounds reported in section 9.8.5.1. They are to be reported in the SDG narrative or a separate form as alkanes by class (straight-chain, branched or cyclic; as a series; as applicable).

9.8.5.6. Quantitative analysis for non-target compounds will be performed using a modified version of the calculation used for target analytes. Non-Target concentrations are calculated using total ion areas for the analyte and the internal standard, and the response factor for the analyte is assumed to be 1.0.

9.8.5.7. The resulting concentration of the non-target compound is flagged to indicate: (a) that the value is an estimate, and (b) which internal standard was used to determine the concentration. Use the nearest internal standard free of interference.

9.8.6. Rounding rule

9.8.6.1. The CLP rounding rule has been changed beginning with this SOW.

9.8.6.2. The new rounding rule stipulates that if the number to be dropped is 5 or greater, then the number retained should be increased by 1.

9.8.6.3. Based on this, all CRQL values for VOC under the "Med. Soil ug/Kg" column in Exhibit C should be changed from 1200 to 1300.

10. Quality Control

10.1. Instrument tuning

10.1.1. Instrument tuning must be evaluated at the beginning of each 12 hour period with the analysis of 50ng of BFB. Ion abundance criteria in Table 3 must be achieved before proceeding to the calibration steps.

10.1.2. All samples, blanks, and QC samples must be analyzed within 12 hours of the injection time of a successful BFB.

10.2. Initial calibration

10.2.1. An initial calibration must be performed to demonstrate instrument sensitivity and linearity prior to sample analysis.

10.2.2. Any time the ion source is cleaned, or other instrument maintenance is done, a new initial calibration is required.

10.2.3. A separate initial calibration is required for ambient purge (for waters and medium level soils), and for heated purge (for low-level soils). The initial calibration for low-level soils must be prepared according to the same preparation method as will be used for the samples.

10.2.4. The initial calibration must contain all of the target analytes and system monitoring compound at the 5 concentration levels.

10.2.5. Calculate a relative response factor (RRF) for each analyte at each concentration level by tabulating peak area response relative to the internal standard against concentration.

10.2.6. Calculate the relative standard deviation (RSD) of the RFs and average RF for each analyte.

10.2.7. Evaluate all compounds for the minimum response factor and maximum RSD criteria listed in Table 7.

10.2.8. Up to two compounds may fail, provided they have a minimum response factor of 0.010 and a RSD less than or equal to 40%.

10.2.9. When calibrating for Xylenes, use the response of the single Xylene isomer (o-Xylene), since the m&p isomers co-elute. This in effect will give identical response factors for all three Xylene isomers.

10.3. Continuing calibration

10.3.1. The initial calibration must be verified every 12 hours with the analysis of a 50ug/L calibration standard.

10.3.2. Calculate a RF for each compound in the continuing calibration standard.

10.3.3. Calculate the %D of the RF 's from the continuing calibration versus the average RFs from the initial calibration.

10.3.4. Evaluate all compounds for the minimum response factor and maximum %D criteria listed in Table 7.

10.3.5. Up to two compounds may fail, provided they have a minimum response factor of 0.010 and a RSD less than or equal to 40%.

10.4. Blank analysis

10.4.1. Method blank

10.4.1.1. A method blank is analyzed once every 12 hours. The method blank is analyzed immediately after the calibration and before any samples are analyzed.

10.4.1.2. The method blank must be prepared in the same manner as the samples, and use the same sample introduction technique.

10.4.1.3. The method blank must not have any target analytes above the CRQL with the following exceptions: methylene chloride and cyclohexane may be up to 2.5 times the CRQL, and acetone and 2-butanone may by up to 5 times the CRQL.

10.4.1.4. The method blank must meet QC limits for acceptable system monitoring compound recoveries.

10.4.1.5. The method blank must meet QC criteria for internal standard response and retention time.

10.4.2. Instrument blank

10.4.2.1. An instrument blank must be analyzed immediately after any sample that has a target compound that exceeds the calibration range.

10.4.2.2. The instrument blank must be analyzed in the same autosampler position as the saturated sample.

10.4.2.3. The instrument blank must meet all criteria for the method blank.

10.4.3. Storage blank

10.4.3.1. A storage blank is prepared with each SDG and will remain with the samples until all have been successfully analyzed.

10.4.3.2. The storage blank must meet all criteria for the method blank.

10.5. System monitoring compounds

10.5.1. System monitoring compound solution containing 1,2-Dichloroethane-d4, Toluene-d8, and Bromofluorobenzene is spiked

into every blank, sample and QC sample at a concentration of 50ug/L.

10.5.2. System monitoring compound recoveries are calculated for all blanks, samples, and QC samples as follows:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

10.5.3. System monitoring compound recovery limits are listed in Table 8.

Table 8 : System monitoring compound recovery limits

Compound	Water limits (% recovery)	Soil limits (% recovery)
1,2-Dichloroethane-d4	76-114	70-121
Toluene-d8	88-110	84-138
Bromofluorobenzene	86-115	59-113

10.5.4. Any blank, sample, or QC sample that fails to meet the criteria must be re-analyzed.

10.5.5. If the re-analysis of a sample has acceptable recoveries, only report data from the second run.

10.5.6. If the re-analysis confirms the failure, report both sets of data, naming the second with a "RE" file extension.

10.5.7. If the sample chosen for MS/MSD fails, and the MS/MSD confirm the failure, no re-analysis is required.

10.6. Internal standard areas and retention times

10.6.1. Internal standard spiking solution containing Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5 is spiked into every blank, sample, and QC sample at a concentration of 50ug/L.

10.6.2. All standards, blanks, samples, and QC samples are monitored for internal standard area response and retention time.

10.6.2.1. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass

spectrometer system must be inspected for malfunction and corrections made as appropriate.

10.6.2.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid-point standard of the initial calibration or the continuing calibration standard.

10.6.2.3. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

10.6.2.4. If the re-analysis of a sample meets criteria, only report data from the second run.

10.6.2.5. If the re-analysis confirms the failure, report both sets of data, naming the second with a "RE" file extension.

10.6.2.6. If the sample chosen for MS/MSD fails, and the MS/MSD confirm the failure, no re-analysis is required.

10.7. Matrix spike/matrix spike duplicate

10.7.1. Matrix spike/matrix spike duplicate pairs are analyzed at a frequency of one set per matrix per SDG.

10.7.2. Water rinsate blanks (field QC) are not to be used for MS/MSD analysis.

10.7.3. Samples must be spiked with 1,1-Dichloroethene, Trichloroethene, Benzene, Toluene, and Chlorobenzene at a concentration of 50 ug/l.

10.7.4. Prepare the MS/MSD for aqueous samples by adding 10 ul of 25 ug/ml matrix spiking solution to each of two 5 ml aliquots of the sample chosen.

10.7.5. Prepare the MS/MSD for low level soils by adding 10 ul of 25 ug/ml matrix spiking solution to the 5 ml of reagent water added to the 5 g aliquots of soil.

10.7.6. Prepare the MS/MSD for medium level soils by extracting 4 g of soil with 9 ml methanol plus 1 ml of 25 ppm spiking solution.

Analyze 100 ul of this extract by adding it to 5 ml reagent water containing internal standard and system monitoring compounds.

10.7.7. Recovery limits and RPD limits for each matrix are listed in Table 9. These limits are advisory only, however frequent failures may indicate a more serious problem.

Table 9: Matrix Spike Recovery and RPD Limits

Compound	Water recovery limits (%)	Water RPD limits	Soil recovery limits (%)	Soil RPD limits
1,1-DCE	61-145	14	59-172	22
TCE	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

11. WASTE MANAGEMENT AND POLLUTION PREVENTION

11.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

11.2. POLLUTION PREVENTION:

11.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When

wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

11.2.2 The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

12. CALCULATIONS

12.1 Refer to the SOP for Organic Calculations, SOP Number OC03.

13. DEFINITIONS

13.1 Refer to document DEFDOC-04 for definitions.

14. METHOD PERFORMANCE

14.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

14.2 All MDL results are available on file.

15. DATA ASSESSMENT AND CRITERIA AND CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

15.1 Technical acceptance criteria for sample analysis.

15.1.1 The samples must be analyzed on a GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.

15.1.2 The sample must be analyzed within the required holding time.

15.1.3 The sample must have an associated method blank meeting the blank technical acceptance criteria.

15.1.4 The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.

15.1.5 The retention time shift for each of the internal standards must be within +/- 0.50 minutes (30 seconds) between the sample and the most recent continuing calibration standard analysis.

15.1.6 After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

15.2 Corrective Action for Sample Analysis

15.2.1 Samples must meet technical acceptance criteria before reporting data.

15.2.2 Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

15.2.3 Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

15.3 If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria:

15.3.1 Check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria

15.3.2 Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

15.3.3 Verify that the instrument is operation correctly.

16. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

16.1 Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

17. REFERENCES

17.1 USEPA Contract Laboratory Program, Statement of Work for Organics Analysis, Multi-Media, Multi-Concentration, Revision OLM04.2, May 1999.

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STL NORTH CANTON STANDARD OPERATING PROCEDURE

TITLE: PREPARATION AND ANALYSIS OF INORGANIC ARSENIC SPECIES IN AQUEOUS SAMPLES, SOILS AND SEDIMENTS BY ANODIC STRIPPING VOLTAMMETRY, MODIFIED METHOD 7063

(SUPERSEDES: REVISION 1, REVISION DATE 02/19/03)

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Proprietary Information Statement:

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LIST OF APPENDICES:

1. SCOPE AND APPLICATION

1.1. This procedure describes the preparation and analysis of arsenic (III) - arsenite [CAS # 22569-72-8] and arsenic (V) – arsenate [CAS # 17428-41-0] by Anodic Stripping Voltammetry (ASV) using Method 7063 modified with techniques and procedures from Trace Detect, Electric Power Research Institute Report EA 4641 and EPA Method 1632A.

1.2. The associated LIMs method code is RM (Method 7063-mod). The sample preparation code for water samples is GW and soil and sediment samples is GX

1.3. ASV analysis provides for the electrochemical determination of dissolved inorganic arsenic (III) and arsenic (V). Detection limits, sensitivity and optimum concentration ranges for arsenic analysis will vary with the matrices, instrumentation and volume of sample used.

1.4. This SOP is applicable to the preparation and analysis of arsenic (III) and arsenic (V) in ground water, surface water and extracts of soil and sediment. Aqueous samples may require filtration, centrifugation, or solid phase extraction cleanup. All solid matrices require sample preparation prior to analysis.

1.5. The STL North Canton reporting limits for arsenic (III) or arsenic (V) in aqueous matrices are 2 ug/L and 100 ug/kg in solid matrices.

2. SUMMARY OF METHOD

2.1. This SOP describes a technique for the determination of inorganic arsenic in aqueous samples and aqueous leachates of solid samples. The procedure is an electrochemical method based on the plating of arsenic (III) at – 400 mV and stripping at + 250 mV. A representative portion of a solid sample is first extracted with an acidic solution (H_3PO_4) and then with a basic solution (Na_3PO_4). The extracts are then combined and analyzed like water samples. The aqueous solution is acidified to 2M with concentrated HCl. A gold film electrode is immersed in the stirred sample aliquot. The plating potential is set to – 400 mV and arsenic (III) is plated on the surface of the gold electrode. The potential is then swept positive and arsenic strips off the electrode around 250 mV. The height and area of the stripping peak is proportional to the concentration of arsenic (III). Since the electrode

sensitivity is affected by non-analyte components of many samples, the concentration is determined using the method of standard additions.

Total inorganic arsenic (As(III) + As(V)) is determined by first reducing the arsenic (V) in the aqueous solution to arsenic (III) with sodium thiosulfate followed by analysis for arsenic (III). Arsenic (V) is then calculated by difference between the total inorganic (III + V) result and the arsenic (III) result.

3. DEFINITIONS

3.1. Inorganic arsenic (III): Those arsenic (III) species soluble in aqueous solutions under the acidic and basic conditions described in this SOP. This does not include insoluble arsenic forms such as sulfides. Also, it does not include organo-arsenicals even when in the +3 oxidation state.

3.2. Inorganic arsenic (V): Those arsenic (V) species soluble in aqueous solutions under the acidic and basic conditions described in this SOP. This does not include insoluble arsenic forms such as sulfides. Also, it does not include organo-arsenicals even when in the +5 oxidation state.

4. INTERFERENCES

Chemical and physical interferences may be encountered when analyzing samples using this method.

4.1. Antimony and bismuth are known interferences because they have stripping potentials similar to arsenic. There are no cleanup procedures or method modifications other than dilution to address this interference at present.

4.2. Copper has a stripping potential near arsenic. If the copper peak is more than twice the size of the arsenic peak some interference may result. The copper may be removed by solid phase extraction with a Copper Remover cartridge (Trace Detect). This cartridge also appears to remove other undefined interferences that reduce electrode sensitivity. Do not use except when needed since some arsenic is also removed.

4.3. High concentrations of non-polar organic compounds can foul the electrode surface and reduce electrode sensitivity. Solid phase extraction with C18 can remove many of these interferences.

4.4. Interfering sample components other than those listed above have been observed but not identified. Sample dilution may be used as needed to maintain electrode performance.

4.5. High concentrations of arsenic (III) can interfere with quantitation of low concentrations of arsenic (V) since arsenic (V) is calculated by the difference of total inorganic arsenic (III + V) and arsenic (III).

4.6. Laboratory contamination may also arise from impure reagents, dirty glassware, improper sample transfers, dirty work areas, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.

4.6.1. Phosphoric acid used to prepare the solid leaching solutions may contain antimony. If the concentration is high enough to cause interference with arsenic quantification, the antimony may be removed with the Copper Remover cartridge.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual and this document.

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Iodine	Poison Corrosive Oxidizer	0.1 ppm-Ceiling	Vapors severely irritate and can burn the mucous membranes and respiratory tract. Liquid contact may cause blistering burns, irritation, and pain. Vapors may be severely irritating to the skin. Vapors are severely irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.3. Arsenic is a highly toxic element that must be handled with care. The analyst must be aware of the handling and clean up techniques before working with arsenic.

5.4. Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Cut resistant gloves must be worn doing any other task that presents a strong possibility of getting cut. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.5. Exposure to hazardous chemicals must be maintained **as low as reasonably achievable.** Therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation where possible. All samples with pink stickers that read "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a STL North Canton associate. The situation must be reported **immediately** to the EH&S Coordinator and to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Anodic Stripping Voltammetry system equipped with:

6.1.1. Gold electrode, Trace Detect Nano-Band or equivalent.

6.1.2. Auxiliary electrode.

6.1.3. Reference electrode, (Ag/AgCl)

6.1.4. Electrode holder.

6.1.5. ASV electronic control instrument and software, Trace Detect Explorer or equivalent.

6.1.6. Computer and printer.

6.1.7. Magnetic stir plate and fluoropolymer coated stir bars.

6.2. Electrode preparation kit

6.2.1. Felt pad on glass plate, one for alumina, one for water, Buehler 40-7722 Microcloth Supreme Final Polishing Cloths, PSA backed.

6.2.2. Alumina polish, Buehler 40-6325-008 Micropolish II, 0.05 micron, Gamma.

6.2.2.1. Combine equal volumes of reagent water and alumina polish in a squirt bottle to make a polish solution.

6.3. Mechanical pipettes and tips: various volumes from 5 uL to 20 mL.

6.4. Disposable gloves, latex or nitrile.

6.5. Disposable beakers, 150 mL and 20 mL.

6.6. Solid phase extraction cartridges or disks

6.6.1. Copper Remover, Trace Detect or equivalent

6.6.2. C18

6.7. Membrane filter, 0.45 um

6.8. Wash bottle, for electrode rinsing with reagent water.

7. REAGENTS AND STANDARDS

7.1. Reagent water must be produced by a deionized water system or equivalent. Reagent water must be free of arsenic and interferences as demonstrated through the analysis of method blanks.

7.2. Stock (1000 mg/L) arsenic (III) standards (in 2% HCl) are purchased. All standards must be stored in previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.3. Stock (1000 mg/L) arsenic (V) standards are purchased. All standards must be stored in previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.4. MSA Working arsenic (III) standard (1000 µg/L): Fill a 50 mL volumetric container about half full with reagent water. Add 1 mL of concentrated HCl. Add 50 µL of stock standard solution (Section 7.2) and dilute to 50 mL with reagent water. The MSA working arsenic (III) standard should be replaced monthly unless studies show stability over a longer period of time. The MSA working standard is used for the method of standard additions, laboratory control sample and matrix spike samples.

Note: Alternate approaches to standard preparation may be taken and alternate volumes of standard may be prepared as long as the accuracy and final standard concentrations are maintained.

7.5. The MSA verification arsenic (III) standard must be made from a different manufacturer or lot than that of the MSA working standard. The MSA verification arsenic (III) standard should be replaced monthly unless studies show stability over a longer period of time.

7.6. Matrix spike arsenic (V) standard (1000 µg/L): Fill a 50 mL volumetric container about half full with reagent water. Add 1 mL of concentrated HCl. Add 50 µL of stock standard solution (Sec. 7.3) and dilute to 50 mL with reagent water. The matrix spike arsenic (V) standard should be replaced monthly unless studies show stability over a longer period of time. The matrix spike arsenic (V) standard is used for the laboratory control sample and matrix spike samples when arsenic (V) is the analyte.

7.7. Hydrochloric acid (HCl), concentrated, trace metal grade.

7.8. Gold stock solution (1000 mg/L) purchased from commercial supplier.

7.9. Electrode plating solution (Au 20 mg/L): 1 mL of gold stock solution (Section 7.8) diluted to 50 mL with reagent water. This reagent should be replaced monthly unless studies show stability over a longer period of time.

7.10. Sodium hydroxide, reagent grade

7.11. Sodium thiosulfate, 0.6% solution.

7.12. Starch, 1.0% solution, Lab Chem LC25310-1 or equivalent.

7.13. Iodine, stock 0.028N solution, Mallinkrodt H169 or equivalent, store in the dark.

7.14. Iodine, working 0.00028N solution, 0.5 mL of iodine stock solution diluted to 50 mL, store in the dark, prepare monthly or sooner if blue color produced with starch looses intensity.

7.15. Phosphoric acid, (H_3PO_4), concentrated, ACS reagent grade, low in arsenic and antimony.

7.15.1. Acidic extraction solution: 0.1 M H_3PO_4, dilute 6.8 mL concentrated phosphoric acid to 1 L with reagent water.

7.15.2. Basic extraction solution: 0.1 M Na_3PO_4, dilute 6.8 mL concentrated phosphoric acid and 12.0 g sodium hydroxide to 1 L with reagent water.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding time for arsenic (III) and arsenic (V) is 28 days from time of collection to the time of analysis if the sample is preserved immediately after collection.

8.2. Aqueous samples are filled to the top of the plastic container with minimal headspace, acidified with HCl to pH <2 and maintained at 4°C ± 2°C from time of collection until analysis.

8.3. Soil and sediment samples are sealed in a glass container and maintained at 4°C ± 2°C from time of collection until analysis.

9. QUALITY CONTROL

9.1. Table III (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.2. Initial Demonstration of Capability

9.3. Prior to the analysis of any analyte using, the following requirements must be met.

9.3.1. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDLs must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below both the STL North Canton reporting limit.

9.3.2. Initial Demonstration Study (initial precision and recovery study)- This requires the analysis of four QC check samples. The QC check sample is a well-characterized laboratory generated sample used to monitor method performance. The results of the initial demonstration study must be acceptable before analysis of samples may begin.

9.3.2.1. Four aliquots of the check sample (LCS) are prepared and analyzed using the procedures detailed in this SOP and the determinative SOPs.

9.4. Preparation Batch - A group of up to 20 samples that are of the same matrix and are processed together using the same procedures and reagents. The preparation batch must contain a method blank, a LCS and a matrix spike/matrix spike duplicate. In some cases, at client request, it may be appropriate to process a matrix spike and sample duplicate in place of the MS/MSD. If clients specify specific samples for MS/MSD, the batch may contain multiple MS/MSD pairs.

9.5. Sample Count - Laboratory generated QC samples (Method Blanks, LCS, and MS/MSDs) are not included in the sample count for determining the size of a preparation batch.

9.6. Method Blank (MB): One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20 times higher than the blank contamination level).

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).
- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**
- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

9.7. Laboratory Control Sample (LCS): One LCS must be processed with each preparation batch. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines. The LCS must be carried through the entire analytical procedure. If the LCS is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, limits of 25- 130% recovery will be applied.

- In the instance where the LCS recovery is greater than the maximum and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- In the event that an MS/MSD analysis is not possible, a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.8. Matrix Spike/Matrix Spike Duplicate (MS/MSD): One MS/MSD pair must be processed for each 20 samples in preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Table I (Appendix A).

- If analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Until in-house control limits are established, a control limit of 25 - 130 % recovery and 50% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits, corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results, which fall outside the control limits, must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4 times the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume, then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

9.9. MSA Verification Standard (MSAV): MSA working standard accuracy is verified by analyzing a second source standard at the same concentration. The MSA verification

standard result must fall within 90-110% of the response for the MSA working standard. An ICB is analyzed immediately following the MSAV to monitor system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the MSAV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected and the instrument recalibrated. (See Section 11.4.4 for required run sequence).

9.10. Method of Standard Addition (MSA) -This technique involves successively spiking known amounts of standard into an aliquot of the sample prior to analysis. This technique compensates for a sample interferent that may enhance or depress the analyte signal. A single point spike at the RL is used for all non-detect samples. Three point MSA is used for all samples with analyte above the RL. Refer to Table II and Section 11.4 for specific MSA guidance.

10. CALIBRATION AND STANDARDIZATION

10.1. Many sample components have small but significant effects on the sensitivity of the gold electrode. There are no clean-up techniques currently available that sufficiently remove all these interferences. Thus, the method of standard additions (MSA) is used to quantify all sample results above the reporting limit. Since MSA used for all quantitative measurements no initial calibration is warranted.

10.2. Condition the gold electrode

10.2.1. Squirt a few milliliters of (1:1) alumina – water mixture (Sec. 6.2.2.1) onto a polishing pad (Sec 6.2.1).

10.2.2. Hold the gold electrode near the base and gently move it in a figure 8 on the polishing pad for about 1 minute. The pressure applied should correspond to approximately 50 g if measured on a top loading balance.

10.2.3. Rinse the electrode thoroughly with reagent water.

10.2.4. Squirt a few milliliters of reagent water onto the water-only polishing pad.

10.2.5. Hold the gold electrode near the base and gently move it in a figure 8 on the polishing pad for about 1 minute. The pressure applied should correspond to approximately 50 g if measured on a top loading balance.

10.2.6. Rinse the electrode thoroughly with reagent water.

10.2.7. Place gold electrode in the three-electrode holder and connect to the instrument. Rinse all electrodes.

10.2.8. Transfer gold electrode plating solution (Sec.7.9) to a plastic beaker. Add stir bar. Immerse electrodes in the solution. Turn on stirring to 75% of maximum.

10.2.9. Load gold plating parameters through the Settings menu. See Table IV.

10.2.9.1. Set plate time to 5 seconds. Click single. Current should be less than 150 nA. If not start over at Sec. 10.2.

10.2.9.2. Set plate time to 55 seconds. Click single. When the measurement is completed the current should less than 200 nA. If not start over at Sec. 10.2.

10.3. Check gold electrode sensitivity

10.3.1. Prepare 1 ug/L sensitivity check standard by combining: 8 mL concentrated HCl, 42 mL reagent water and 50 uL of 1000ug/L As standard (either Sec. 7.2 or 7.5). Add stir bar.

10.3.2. Rinse electrodes with reagent water then immerse in the 1 ug/L solution. Turn on stirring to 75% of maximum.

10.3.3. Load arsenic analysis parameters through the Settings menu. See Table IV.

10.3.4. Set plate time to 160 seconds. Click single. When the measurement is completed the arsenic peak height (@~250 mV) must be at least 5 times the background noise. Generally peak height is between 50 to 100 nA with noise height of about 5 nA. If not, repeat the measurement. If the signal to noise ratio is still too low, recondition or replace the gold electrode.

10.4. Check MSA working standard

10.4.1. Prepare a 20 ug/L MSA working standard solution by combining: 8 mL concentrated HCl, 42 mL reagent water and 1000 uL of MSA working standard (Sec. 7.2). Add stir bar.

10.4.2. Prepare a 20 ug/L MSA verification standard solution by combining: 8 mL concentrated HCl, 42 mL reagent water and 1000 uL of MSA verification standard (Sec. 7.5). Add stir bar.

10.4.3. Immerse the electrodes in the working standard solution (Sec. 10.4.1). Set plate time to 40 seconds. Click continuous. After 2-3 scans the peak height should be consistent. Click cancel. Record the As peak height. Rinse electrodes.

10.4.4. Repeat the measurement with the verification standard solution (Sec. 10.4.2). Compare the working standard and verification standard results with the acceptance criterion in Table III.

10.5. Check system cleanliness

10.5.1. Prepare a system blank solution by combining: 8 mL concentrated HCl, 42 mL reagent water. Add stir bar.

10.5.2. Set plate time to 160 seconds. Click single. When the measurement is completed the arsenic peak height (@~250 mV) must be less than 50% of that recorded for the 1 ug/L sensitivity check in Sec. 10.3.4. Typically there is no detectable As peak. If too much arsenic is present examine the electrode cleaning process or reagents. Correct the problem and repeat the test. If a reagent change was necessary repeat the sensitivity check as well.

11. PROCEDURE

11.1. Water Sample Preparation:

11.1.1. Combine 8 mL of concentrated HCl with 42 mL of sample. Add 0.84 mL of MSA working standard (Sec. 7.2) if this is a LCS, MS or MSD for arsenic (III). Or use 0.84 mL of matrix spike arsenic (V) standard (Sec. 7.6) for arsenic (V) analyses. Either standard produces a true spike concentration of 20 ug/L for the respective arsenic species.

11.1.2. If prior knowledge or preliminary screen indicates the presence of a Cu or other interferences then allow 50- 100 mL of acidified sample to drip through Copper Remover SPE cartridge (Sec. 6.6.1) at a rate of about 50 mL/hr.

11.1.3. Use C18 SPE (Sec. 6.6.2) to remove non-polar interferences as needed.

11.1.4. Add stir bar.

11.1.5. If the sample is to be tested for total inorganic arsenic (III + V) reduce the arsenic (V) to arsenic (III)

11.1.5.1. Add 100 uL of sodium thiosulfate reducing solution (Sec. 7.11) to each 50 mL sample and stir.

11.1.5.2. Wait at least 10 minutes at room temperature.

11.1.5.3. Test for excess reducing agent to ensure that the sample matrix has not consumed all the thiosulfate.

11.1.5.3.1. Put 200 uL of starch indicator (Sec. 7.12) into a small disposable beaker. Add 50 uL of the working iodine solution (Sec. 7.14) and mix gently.

11.1.5.3.2. Add 200 uL of reduced sample directly to the blue starch – iodine mix. If the blue color dissapates imediately then excess reducing agent is present. If the blue color fades because of dilution with the sample water but does not dissapear then the thiosulfate has been consumed by oxidizers in the sample.

11.1.5.3.3. Add more thiosulfate if needed. Allow 10 minutes reaction time and retest for excess reducing agent.

11.1.6. Proceed to Sec. 11.3 or Sec. 11.4 for screening or analysis.

11.2. Soil & Sediment Sample Preparation:

11.2.1. Weigh 8 g of solid into 250 mL plastic bottle. Record weight. Add 8 uL of stock arsenic (III) standard (Sec. 7.2) if this is a LCS, MS or MSD for arsenic (III). Or use 8 uL of stock arsenic (V) standard (Sec. 7.3) for arsenic (V) analyses. Either standard produces a true spike concentration of 1000 ug/kg for the respective arsenic species.

11.2.2. Add 200 mL of acidic extraction solution (Sec. 7.15.1) and shake.

11.2.3. Allow the mixture to sit at room temperature for about two hours, shaking periodically. Refrigerate overnight at 4°C ± 2°C.

11.2.4. Decant into a different plastic bottle. Add 200 mL of basic extraction solution (Sec. 7.15.2) and shake.

11.2.5. Allow the mixture to sit at room temperature for about two hours, shaking periodically. Refrigerate overnight at 4°C ± 2°C.

11.2.6. Combine 8 mL of conc. HCl, 20 mL aliquot of acidic extract and 20 mL aliquot of basic extract.

11.2.7. Process as a water sample from this point starting at Sec. 11.1.2.

11.3. Sample screening:

11.3.1. Set plate time to 5 sec. Click single. Assess arsenic concentration and potential interferences. Increase plate time plate time as needed. See Table II for recommended plate times for various concentrations.

11.3.2. Perform cleanups and dilution as needed (Sections, 11.1.2, 11.1.3).

11.4. Sample Analysis:

11.4.1. Based on the screening results select the plate time that will produce an arsenic signal to noise ratio of about 50. See Table II for guidance. If the arsenic concentration is below 5 ug/L set the plate time to a maximum of 160 sec. If the arsenic concentration is > 500 ug/L dilute the sample prior to analysis.

11.4.2. If the sample is non-detect with a 160 sec. plate time, add 50 uL of a 1000 ug/L As (III) standard (either Sec. 7.2 or 7.5) and reanalyze with 160 sec plate time.

11.4.2.1. This 1 ug/L As (III) peak should be clearly visible and have a signal to noise ratio greater than 5. Print the scans that show both the original sample and the single point MSA that documents the non-detect. Figure 1 shows the screen display prior to printing.

11.4.2.2. If the As (III) spike peak is small or non-existent one of three scenarios is likely:

11.4.2.2.1. The copper peak (@~450 mV) is over-riding the arsenic peak. Take a fresh aliquot of sample or sample extract and proceed with the copper removal clean-up (Sec. 11.1.2).

11.4.2.2.2. The sample contains interferences that have fouled the electrode surface and reduced its sensitivity. Take a fresh aliquot of the sample or sample extract and use either the copper removal cleanup (Sec. 11.1.2) or C18 SPE clean-up (Sec. 11.1.3) or dilute to reduce the interferences such that the 1 ug/L single point MSA produces an acceptable peak.

11.4.2.2.3. The sample contains an oxidizer that is converting arsenic (III) to arsenic (V), which is not recovered under these instrument settings. Spike the sample aliquot with an additional 20 ug/L arsenic (III). Stir the sample for 2 minutes and reanalyze. If an oxidizer is present the arsenic peak with be much smaller than expected or non-detect. Print out all scans to demonstrate the loss of arsenic (III).

11.4.2.3. Reanalyze the electrode sensitivity check standard (1 ug/L). Absolute peak height must be at least 10 % of the height at the start of the sequence and signal to noise ratio > 5.

11.4.3. If the sample scan shows an arsenic peak that may be greater than the reporting limit, proceed with MSA using a minimum of 3 additions. See Table II.

11.4.3.1. Estimate the sample concentration based on the peak height in the unknown sample, responses from standards earlier in the sequence and other MSA "calibration" data from similar samples.

11.4.3.2. Spike in a concentration approximately equal to that of the original sample. Thus, the peak height should approximately double going from the original sample to the first addition. When in doubt, it is better to underestimate the initial sample concentration and spike too low than over estimate and spike too high. If the first MSA spike was less than 50% of the original sample concentration (i.e. the peak height increase was < 50%), include a 4^{th} addition in the MSA.

11.4.3.3. Successive MSA spikes should increase the total peak height by about the same amount as the original sample peak height.

11.4.3.4. Display and print all MSA scans, addition amounts and "calibration" curves. See Figures 2, 3 and 4. The fit factor (r^2) must be 0.98 or greater or the MSA must be repeated with a fresh sample aliquot.

11.4.4. Example analytical sequence:

Electrode Sensitivity Check (1 ug/L)
MSA Working Standard (20 ug/L)
MSA Verification Standard (20 ug/L)
System Cleanliness Check (ICB)

Sample 1 – non-detect
Sample 1 – single point MSA (1 ug/L)

Sample 2 – As peak present
Sample 2 – 1st MSA addition
Sample 2 – 2nd MSA addition
Sample 2 – 3rd MSA addition

Sample 3 – non-detect
Sample 3 – single point MSA (1 ug/L) – still no As peak
Sample 3 – 20 ug/L addition, wait 2 min – still no As peak –
report ND narrate sample converts As(III) to another As species

Electrode Sensitivity Check (1 ug/L) – peak height and S/N OK

Sample 4 – non-detect (large peak near 450 mV)
Sample 4 – single point MSA (1 ug/L) – still no As peak,
Cu interference suspected,
reprepare sample with Cu removal cleanup

Electrode Sensitivity Check (1 ug/L) – peak height and S/N OK

Sample 5 – non-detect
Sample 5 – single point MSA (1 ug/L) – still no As peak
electrode fouling suspected,
reprepare sample with C18 cleanup or dilution

Electrode Sensitivity Check (1 ug/L) – peak height and S/N too low
put electrodes in mid level standard solution
continuously cycle until sensitivity improves
or recondition electrode

Electrode Sensitivity Check (1 ug/L) – peak height and S/N OK

Sample 6 – 5 second screening shows As concentration too high
dilute and continue with MSA
Sample 6 (diluted) – As peak present
Sample 6 (diluted) – 1st MSA addition
Sample 6 (diluted) – 2nd MSA addition
Sample 6 (diluted) – 3rd MSA addition

11.5. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.6. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Signal to noise ratio (S/N) is calculated according to the equation:

$$S/N = \left(\frac{Ht}{N}\right)$$

Where:
S/N = signal to noise ratio
Ht = Arsenic standard peak height (nA)
N = baseline noise (nA)

12.2. MSA Working Standard check is calculated according to the equation:

$$V\%R = 100\left(\frac{MSA\_Vstd}{MSA\_Wstd}\right)$$

Where:
V %R = percent recovery of verification standard
MSA_Wstd = Arsenic peak height (nA) in MSA working standard
MSA_Vstd = Arsenic peak height (nA) in MSA verification standard

12.3. Method of Standard Addition concentrations are calculated according to the following equations:

The data is fit with a weighted linear regression line. The weighting factors for the individual data points are based on the standard deviation approximation (σ_i) for that point. This

corresponds to the maximum of 3% of the peak height or 5 times the noise $\sigma_i = \mathrm{MAX}[0.03y_i, 5i_{noise}]$.

$$a = \frac{S_{xx}S_y - S_xS_{xy}}{\Delta}, \quad b = \frac{SS_{xy} - S_xS_y}{\Delta}, \quad \Delta = SS_{xx} - (S_x)^2$$

$$S = \sum_{i=1}^{N} \frac{1}{\sigma_i^2} \quad , \quad S_x = \sum_{i=1}^{N} \frac{x_i}{\sigma_i^2} \quad , \quad S_y = \sum_{i=1}^{N} \frac{y_i}{\sigma_i^2} \quad ,$$

$$S_{xx} = \sum_{i=1}^{N} \frac{x_i^2}{\sigma_i^2} \quad , \quad S_{xy} = \sum_{i=1}^{N} \frac{x_i y_i}{\sigma_i^2}$$

a = intercept
b = slope
x = total concentration added
y = peak height

Once the values for intercept (a) and slope (b) are determined for the linear equation $y(x) = a + bx$, the absolute value of the "x" intercept corresponds to the original concentration in the measured sample aliquot.

$C_s = |-a/b|$

C_s = Concentration of sample aliquot

12.4. Matrix spike recoveries are calculated according to the following equation:

$$\%\,R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.5. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:

MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:

DU1 = Sample result
DU2 = Sample duplicate result

12.6. The final concentration for an aqueous sample is calculated as follows:

$$ug/L = C \times D$$

Where:

C = Concentration (ug/L) from instrument readout
D = Instrument dilution factor

12.7. The final concentration for a soil sample is calculated as follows:

$$ug/kg = C \times PR \times D$$

Where:

C = Concentration (ug/L) from instrument readout
PR = Preparation ratio, normally 50, (400 mL to 8 g)
D = Instrument dilution factor

12.8. The arsenic (V) concentration is calculated according to the following equation:

As(V) *ug/L* = As(III+V) *ug/L* - As(III) *ug/L*

As(V) *ug/kg* = As(III+V) *ug/kg* - As(III) *ug/kg*

12.9. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.10. Appropriate factors must be applied to sample values if dilutions are performed.

12.11. Sample results should be reported with up to three significant figures in accordance with the STL North Canton significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.

13.2. Method performance is determined by the analysis of method blanks and laboratory control samples. The method blanks must meet the criteria in Section 9.6. The laboratory control sample should recover within 25 to 130% of the true value until in house limits are established.

13.3. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method allows for the proportional reduction of sample and reagent volumes to decrease waste generation.

15. WASTE MANAGEMENT

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

15.2. The following waste streams are produced when this method is carried out

15.2.1. Expired standards. Working standards are disposed of in the flammable waste containers identified as "Mixed Flammable Solvent Waste" located in the Lab. Concentrated standards are disposed of in Lab packs by the Waste Coordinator.

15.2.2. Laboratory generated aqueous waste. This waste may have a pH of less than 2.0. Waste stream must be collected in a designated container identified as "Acid Waste" and neutralized before discharge to a sewer system if the pH is less than 4.

16. REFERENCES

16.1. References

16.1.1. Method 7063, Arsenic in Aqueous Samples and Extracts by Anodic Stripping Voltammetry (ASV), U.S.EPA, December 1996.

16.1.2. EPA/600/X-93/017, Quantifying Arsenic in Aqueous Solutions by Anodic Stripping Voltammetry, U.S.EPA, February 1993.

16.1.3. Method 1632, Revision A, Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry, U.S. EPA August 1998.

16.1.4. Electric Power Research Institute EA-4641, Speciation of Selenium and Arsenic in Natural Waters and Sediments, Volume 2, Arsenic Speciation, Battelle, Pacific Northwest Laboratories, June 1986.

16.1.5. Corporate Quality Management Plan (QMP), current version.

16.1.6. STL Laboratory Quality Manual (LQM), current version.

16.1.7. STL Corporate Safety Manual, M-E-0001 and STL North Canton Facility Addendum and Contingency Plan, current version.

16.2. Associated SOPs and Policies, latest version

16.2.1. User's Guide, Nano-Band Explorer Version 2.3, Trace Detect, 2001.

16.2.2. QA Policy, QA-003

16.2.3. Glassware Washing, NC-QA-0014

16.2.4. Statistical Evaluation of Data and Development of Control Charts, NC-QA-0018

16.2.5. Method Detection Limits and Instrument Detection Limits, S-Q-003 and NC-QA-0021

16.2.6. Navy/Army SOP, NC-QA-0016

17. MISCELLANEOUS (TABLES, APPENDICES, ETC. . .)

17.1. Modifications/Interpretations from reference methods.

17.1.1. This SOP combines procedures from references 6.1.2, 6.1.3, 6.1.4, 6.2.1.

The key differences between Method 7063 and this SOP are:

17.1.2. Method 7063 claims to determine total inorganic arsenic (As (III) + As(V)). This ASV determination is selective for arsenic (III). Arsenic (V) is chemically reduced to arsenic(III) with sodium thiosulfate to allow the determination of total inorganic arsenic (As (III) + As (V)). Arsenic (V) is then calculated by difference.

17.1.3. Method 7063 uses a gold film glassy carbon electrode. This SOP uses a gold micro array electrode.

17.1.4. Method 7063 uses an initial calibration and periodic calibration verification standards (ICV and CCV). This SOP uses method of standard additions.

17.1.5. Method 7063 uses nitric acid and room temperature storage for water sample preservation with no stated holding time. This SOP uses hydrochloric acid and refrigerated storage with a 28 day holding time for water samples.

17.1.6. Method 7063 contains no information regarding the storage or preparation of solid samples. This SOP includes guidance drawn from other references.

17.2. Modifications from previous SOP

17.2.1. None.

17.3. Facility Specific SOPs

17.3.1. Not applicable.

17.4. Documentation and Record Management

The following documentation comprises a complete raw data package:

- Raw data (direct instrument printouts: ASV scans, MSA curves, MSA additions)
- Run log printout from instrument software. (A bench sheet may be substituted for the run log as long as it contains an accurate representation of the analytical sequence).
- Data review checklist - See Appendix B
- Standards Documentation (source, lot, date).
- Copy of digestion log.
- Non-conformance summary (if applicable).

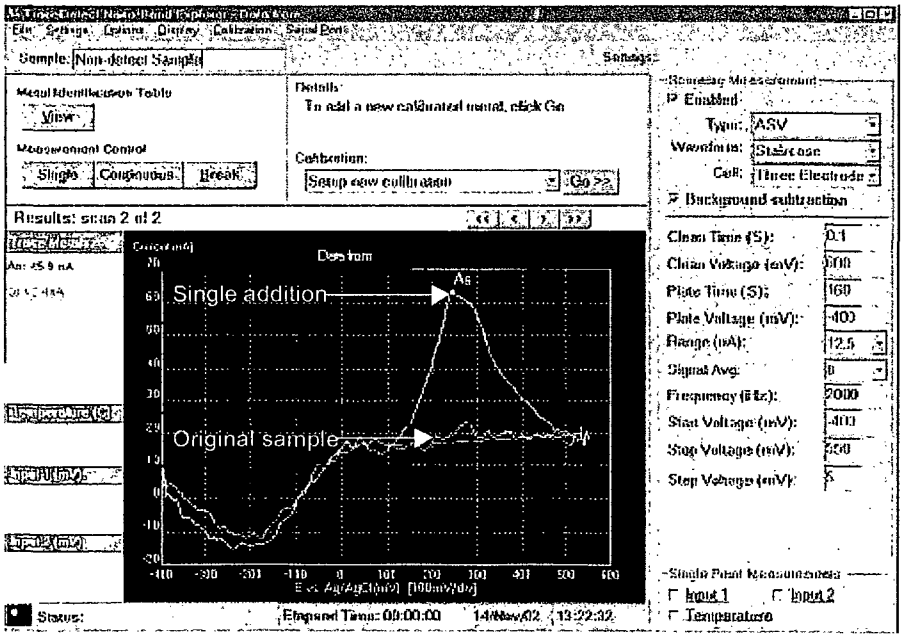

Figure 1. Screen Display of Non-detect Sample with Single Point MSA at 1 ug/L

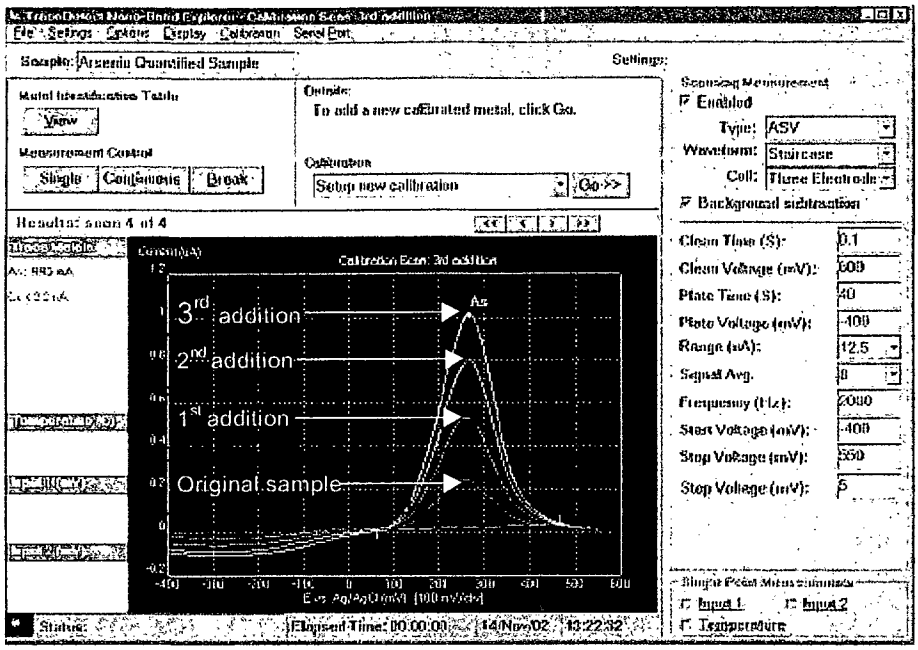

Figure 2. Screen Display of Detect Sample with Three Point MSA.

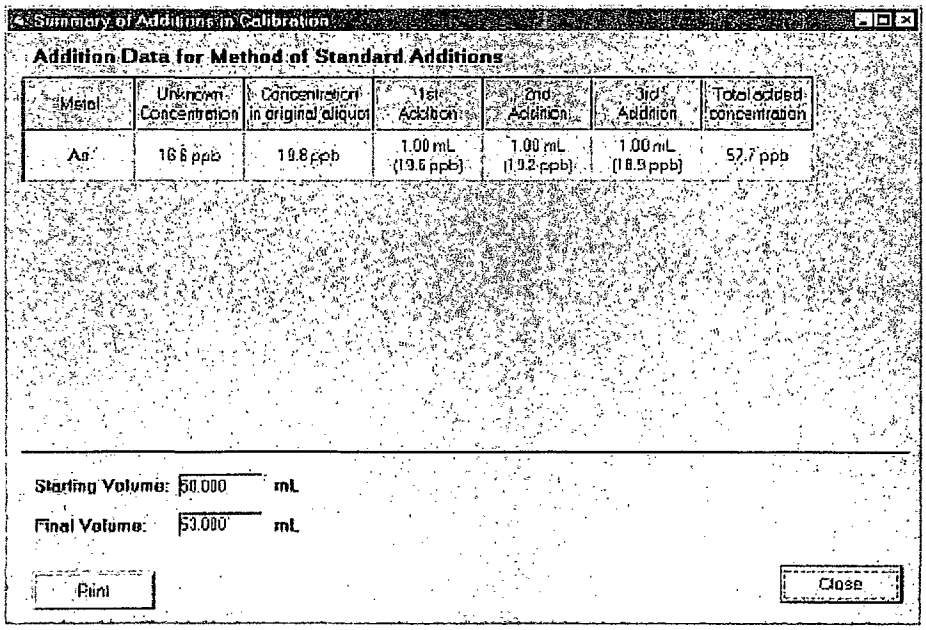

Figure 3. MSA Addition Amounts and Calculated Original Sample Concentration

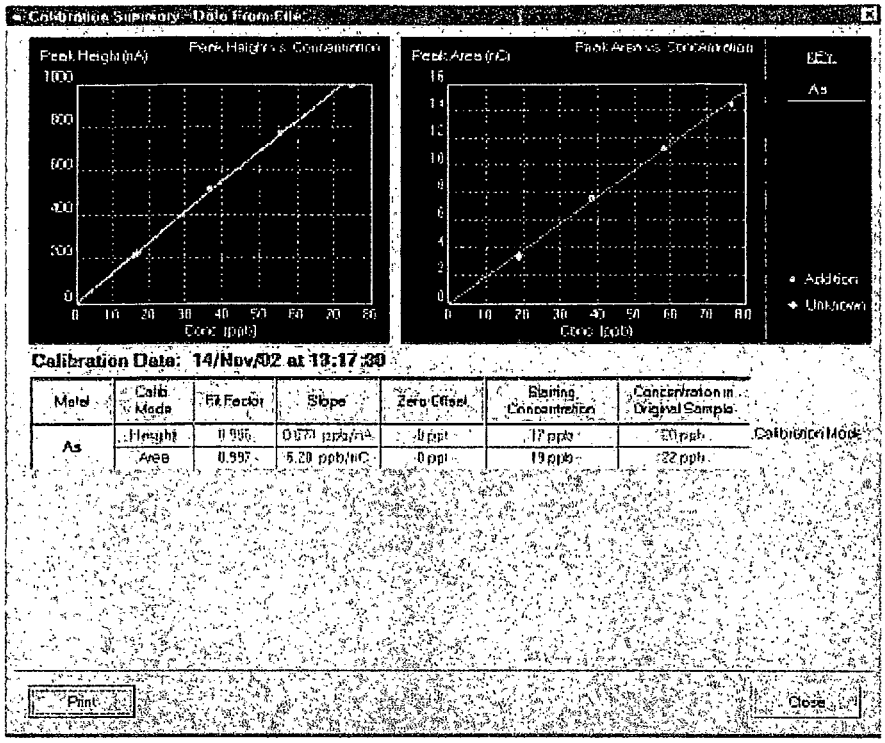

Figure 4. MSA Addition Calibration Curves for Peak Height and Peak Area.

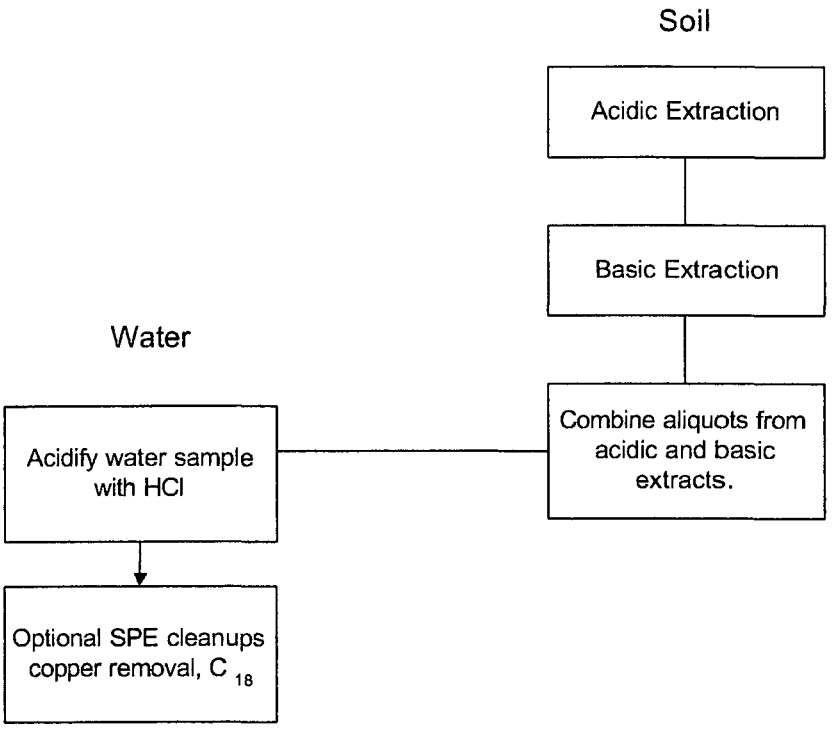

Figure 5. Water and Soil Preparation

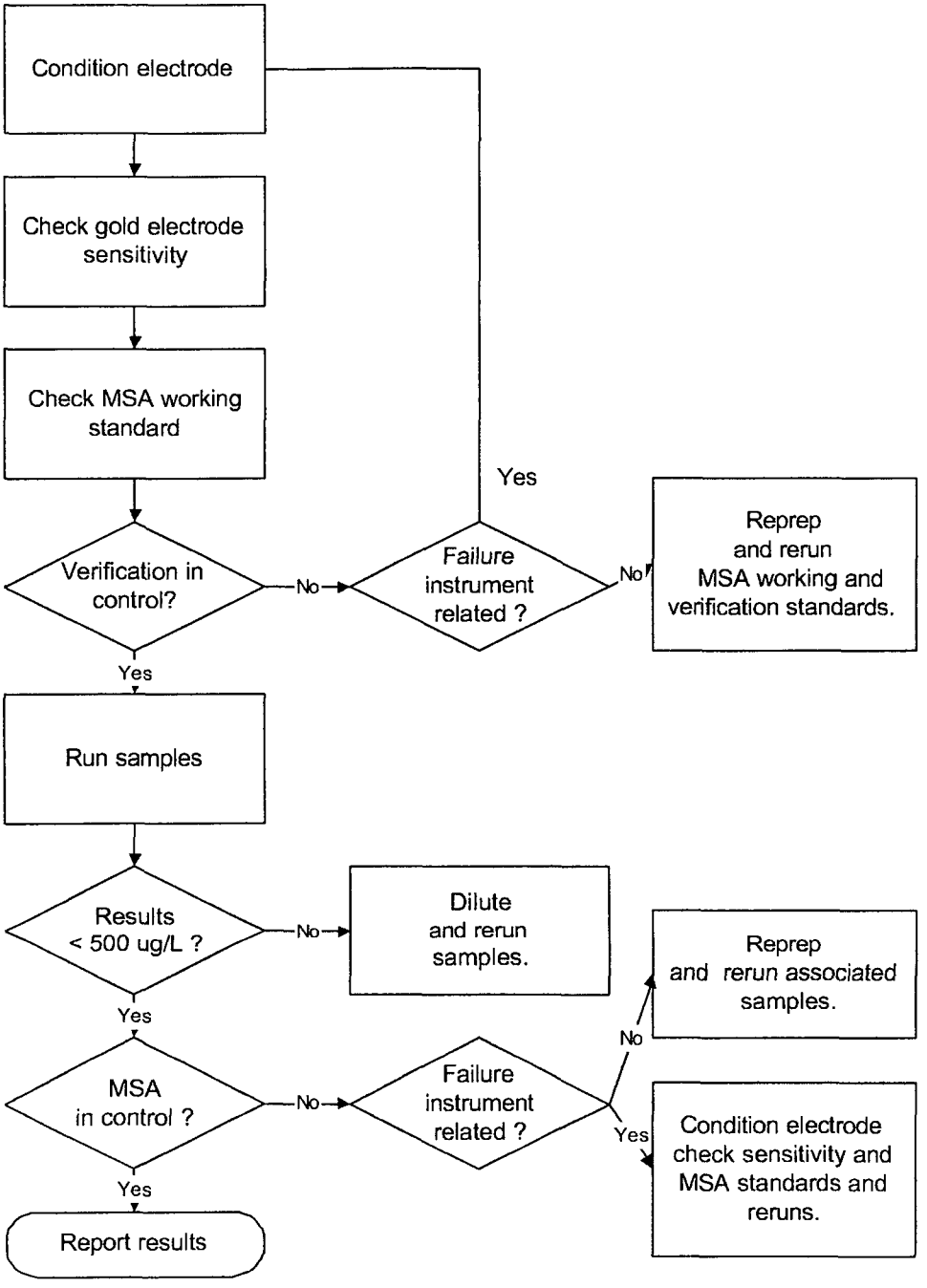

Figure 6. Aqueous Solution Analysis

APPENDIX A

TABLES

TABLE I . ARSENIC (III) REPORTING LIMITS, QC STANDARD AND SPIKING LEVELS

	Water	Soil & Sediment
	Conc ug/L	Conc ug/kg
Standard RL	2	100
MSA working std	20	
MSA verification std	20	
LCS	20	1000
MS	20	1000

TABLE II. Guidance for Method of Standard Addition Spike Levels and Plate Time

Approximate conc (ug/L)*	Plate time (Sec)	Number of scans per MSA reading	MSA Spike concentrations (ug/L)	MSA Spike volumes (uL)**
1 to 5	160	1	1, 2, 4	50, 100, 200
5 to 20	80	1	4, 10, 20	200, 500, 1000
20 to 50	40	2	20, 50	1000, 2500
				MSA Spike volumes (uL)***
50 to 100	20	3	50, 100	2.5, 5
100 to 200	10	3	100, 200	5, 10
200 to 500	5	3	200, 500	10, 25

* in solution to be analyzed

** MSA working standard (Sec.7.2) *** Use stock standard (Sec. 7.2)

TABLE III. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Electrode sensitivity check	Beginning of every analytical sequence, after samples with no MSA recovery and after electrode conditioning	Signal to noise ratio should be >5 for a 1 ug/L standard	Recondition or replace gold electrode
MSA working standard	Beginning of every analytical sequence.	No specific criteria, this is the reference point for the MSA verification standard	
MSA verification standard	Beginning of every analytical sequence.	90-110 % recovery compared to MSA working standard	Terminate analysis; Correct the problem; replace standards as needed. (See Section 9.9).
ICB	Beginning of every analytical sequence, immediately following the MSA verification standard.	The result must be within +/- RL (2 ug/L or 100 ug/kg)	Terminate analysis; Correct the problem; Recalibrate or reprep with calibration curve. (See Section 9.9).

*See Section 1.2 for exact run sequence to be followed.

TABLE III. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA 1631B	CORRECTIVE ACTION
MSA sample calibration	Every sample with arsenic concentration > RL	Correlation coefficient, r > 0.98	Reanalyze sample if MSA spiking error. Use SPE cleanup or dilution if interferences present, then reanalyze.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be within +/- RL (2 ug/L or 100 ug/kg) Sample results greater than 20x the blank concentration are acceptable.	Reprepare and reanalyze samples. Note exceptions under criteria section. See Section 9.6 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	25-130 % recovery	Terminate analysis; Correct the problem; Reprepare and reanalyze all samples associated with the LCS (see Section 9.7).
Matrix Spike	One per sample preparation batch of up to 20 samples.	25-130 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. (see Section 9.8)
Matrix Spike Duplicate	See Matrix Spike	25-130 % recovery; RPD ≤ 50%. (See MS)	See Corrective Action for Matrix Spike.

TABLE IV. Summary of Instrument Parameters (Trace Detect Nano Band Explorer)

Instrument Parameter	Gold plating	Arsenic (III) plating
Type	ASV	ASV
Waveform	square wave	staircase
Cell	3 electrode	3 electrode
Clean time (S)	0	0.1
Clean Voltage (mV)	100	600
Plate Time (S)	5 and 55	5 to 160
Plate Voltage (mV)	-50	-400
Range (uA)	1.25	12.5
Signal Avg	16	8
Frequency (Hz)	1000	2000
Start Voltage (mV)	-50	-400
Stop Voltage (mV)	5	550
Step Voltage (mV)	5	5

APPENDIX B
EXAMPLE
STL NORTH CANTON As(III) DATA REVIEW CHECKLIST

STL North Canton Arsenic (III) Data Review Checklist

Run/Project Information

Run Date: _____ **Analyst:**_____ **Instrument:**_____
Prep Batches Run:_____

Methods used: **7063 mod :** NC-WC-000X Rev 1

Review Items

A. Instrument Setup	Yes	No	N/A	2ndLevel
1. Electrode sensitivity check?				
2. MSA working standard and verification standard within control limits?				
3. Cleanliness check (ICB) within +/- RL?				
B. Sample Results				
1. Were non-detect samples verified with single point MSA?				
2. All samples with As(III) quantified with 3-4 point MSA?				
3. Sample analyses done within holding time?				
C. Preparation/Matrix QC				
1. Samples preserved in the field?				
2. LCS done per prep batch and within QC limits?				
3. Method blank done per prep batch and < RL?				
4. MS run at required frequency (1 per 20 samples) and within limits?				
5. MSD or DU run at required frequency (1 per 20 samples) and RPD within limits?				
D. Other				
1. Are all nonconformances documented appropriately?				
2. Current MDL data on file?				
3. Calculations and Transcriptions checked for error?				
4. All client/ project specific requirements met?				
5. Date of analysis verified as correct?				

Analyst: _____ **Date:**_____
Comments:
_____
_____
_____

2nd Level Reviewer : _____ **Date:**_____
_____
_____
_____

Standard, Reagent and Supply Numbers

MSA Working Std _____ **MSA Verification Std** _____ **MS As(V) Std** _____

Au plate soln _____ **conc HCl** _____ **Cu Remover** _____ **C18**_____

Acid ext _____ **Basic ext** ____

Appendix C ARSENIC SPECIATION LEACHING LOGSHEET

Prep Date _____ Batch _____

Analyst(s) _____ Start Time/Date Acidic:_____Basic:_____

End Time/Date Acidic:_____Basic:_____

Sample	Sample Amount (g)	Acidic Leach Volume (mL) #WR_____	Basic Leach Volume (mL) #WR_____	Comments

APPENDIX D. TROUBLESHOOTING GUIDE

Problem	Possible Cause	Corrective Action
Poor sensitivity	gold electrode not conditioned properly	recondition electrode
	gold electrode surface fouled	multiple cycles in a clean standard recondition electrode
high current during gold plating step	cracked glass plate on gold electrode failed epoxy seal on gold electrode	replace electrode
	gold electrode not conditioned properly	recondition electrode
very high noise in ASV scan	cracked glass plate on gold electrode failed epoxy seal on gold electrode	replace electrode
	gold electrode not conditioned properly	recondition electrode

STL Edison Standard Operating Procedure	Revision Date: 30 March 2004
Title: SW846 7196A, The Analysis of Waters for Hexavalent Chromium	SOP Number C7196AW04
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File Location: f:\qaqc\sops\nelac\nelac sops 2004\140wetch\cr6\c7196aw04.doc	

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1. SCOPE AND APPLICATION:

1.1. SW846 Method 7196A may be used to determine the hexavalent chromium content aqueous samples of aqueous or digestates of solid samples. This SOP is applicable to determination hexavalent chromium of water samples. Solid samples must be first digested according to Method 3060A USEPA SW846 3rd Edition.

1.2. The range of analysis by this method is 10-200 ug/l Cr(VI).

2. METHOD SUMMARY

2.1. Dissolved hexavalent chromium is determined spectrophotometrically at 540nm by reaction with diphenylcarbazide in acid solution. Each batch of samples a matrix spike, matrix spike duplicate, duplicate, blank, laboratory control sample and post digestion spike is analyzed and compared to laboratory control limits.

3. INTERFERENCES

3.1. Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

3.2. Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of Cr(VI) present.

3.3. Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

3.4. Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time, however, interference is checked by post digestion spike samples.

3.4.1. The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

3.5. For waste materials containing soluble Cr(III) concentrations greater than four times the laboratory CR(VI) reporting limit, CR(VI) results obtained using this method may be biased high due to method-induced oxidation.

4. APPARATUS AND MATERIALS

4.1. pH meter, Orion benchtop or equivalent

4.2. Spectrophotometer for use at 540 mm with a light path of 1 cm.(Sequoia Turner)

4.3. Membrane filters, 0.45 um and 0.1 um. (NALGENE disposable)

4.4. Glassware, assorted volumetric pipets (class A), volumetric Flasks (class A), beakers, Erlenmeyer flasks with stoppers.

4.5. Vacuum pump

4.6. Specimen Cups (plastic)

4.7. Magnetic stirrer (teflon-coated)

5. REAGENTS

5.1. Sulfuric acid (10% v/v): Measure 100.0 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water.

5.2. Indicator solution: Place 5.0 g 1,5-Diphenylcarbazide (AR Grade) in an amber 1000ml volumetric flask and dilute to mark with acetone. Prepare monthly or when cloudy.

5.3. Acetone (reagent grade).

5.4. Nitric Acid, concentrated reagent grade

6. STANDARDS

6.1. 1000 mg/L Hexavalent Chromium Standard- purchased from Inorganic Ventures, Lakewood, NJ.

6.2. Hexavalent Chromium Spiking Solution (100 mg/L Cr+6) Prepare by adding 10ml of 1000 ppm Cr+6 Standard to a 100ml volumetric flask and dilute to volume with deionized water. Stable for 6 months.

6.3. Hexavalent Chromium Spiking Solution (10 mg/L Cr+6) Prepare by adding 10.0ml of 100 ppm Cr+6 Standard to a 100ml volumetric flask and dilute to volume with deionized water. Stable for 6 months.

6.4. Potassium dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of dried(105°C) potassium dichromate and dissolve in deionized water in a 100ml flask. Dilute to the mark. Stable for 6 months.

6.5. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10ml of 1000 ppm Cr+6 Secondary Standard to a 100ml volumetric flask and dilute to volume with deionized water. Stable for 6 months.

6.6. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) Prepare by adding 10.0ml of 100 ppm Cr+6 Secondary Standard to a 100ml volumetric flask and dilute to volume with deionized water. Stable for 6 months.

7. PRESERVATION AND HANDLING

7.1. Samples must be analyzed within 24 hours from time of collection. Store samples at 4°C before analysis or digestion.

7.2. For sample homogenization refer to SOP Subspl04.

8. SAFETY

8.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

8.3. All questions pertaining to any safety procedure should be brought to the department manager or Edison Safety Officer.

8.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

8.5. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.

9. PROCEDURE

9.1. Instrument Operating Parameters

9.1.1. Warm up spectrophotometer and set to 540nm.

9.2. Calibration and Calibration Verification

9.2.1. Everyday or before each analysis, prepare fresh calibration standards.

9.2.2. Calibration Standards: (6 points). Place about 25 mls of deionized water in six 50 ml flasks. Prepare 0, 10, 50, 75, 100, and 200 ppb $K_2Cr_2O_7$ solutions by diluting $K_2Cr_2O_7$ solution (use 0.05, 0.25, 0.375, 0.50 and 1.0 mls of 10ppm stock respectively). Bring up to volume with deionized water.

9.2.3. Analyze calibration standards in the same manner as samples. Calibrate, or "zero", the spectrometer with the 0 ppb standard.

9.2.4. Develop the color for the standards as explained in the following Sections of this SOP.

9.2.5. Following the preparation of the calibration standards, "zero" the spectrophotometer with the 0 ppb standard. Analyze the standards.

9.3. Sample Analysis

9.3.1. Place 45.0 mls of sample into a 100 ml specimen cup. Also, place 45 mls of sample in a separate cup for background.

9.3.2. Add 1.0 ml indicator solution and swirl. (Do not add indicator to background sample).

9.3.3. Slowly add 10% H_2SO_4 dropwise to sample and background while measuring pH until pH is 1.6-2.2. Record reading.

9.3.4. Place sample in a 50 ml volumetric flask and bring to volume with deionized water. Pour back into original cup and swirl.

9.3.5. Let stand 5-10 minutes for full color development.

9.3.6. Read calibration standards, QA samples, and samples on spectrophotometer at 540 nm.

9.3.7. Immediately following calibration of the spectrophotometer analyze the calibration check standard and calibration check blank.

9.3.8. Dilute samples that exceed calibration curve using deionized water. Recolorize and read again on spectrophotometer noting dilution factor.

9.3.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

9.3.10. After every 10 samples and after reading the last sample in the batch, verify the spectrometer calibration by reading a calibration check standard and calibration check blank.

9.4. Calculations

9.4.1. Calculate the hexavalent chromium result for each aqueous sample as follows:

9.4.1.1. Hexavalent Chromium in ug/L = A x E

A = Concentration from the calibration curve in ug/L
E = Dilution (if necessary)

9.4.2. Enter values recorded in analysis logbook into the computer under Excel, cr6scal (soil) or cr6wcal (water).

9.4.3. Perform linear regression and verify that the correlation coefficient is greater than 0.997.

9.4.4. Print worksheet, calibration curve and quality control sheet and record results in logbook.

9.5. Data Processing

9.5.1. Transfer results from logbook to analyte sheets and attach computer print-out copies.

9.5.2. Fill out chronicle sheets in job folder and place analyte sheets inside.

9.5.3. Turn QA sheets into manager with printout sheets attached.

10. QUALITY CONTROL

10.1. Midpoint Calibration Check Standard/Calibration Check Blank: Immediately after analyzing the calibration standards, after

every 10 samples (20 readings including backgrounds), and after reading the last sample, analyze a calibration check standard and calibration check blank. A calibration check standard is a separate mid-point calibration standard at 75 ppb Cr(VI). A calibration check blank is a separate 0 ppb calibration standard. The calibration check standard must be 90-110% of the true value. The calibration check blank must remain below the method detection limit.

10.1.1. The following calculation should be used to determine percent recovery of the calibration check standard (CCS).

$$\text{CCS Recovery} = \frac{\text{CCS result}}{\text{True Value}} \times 100$$

10.2. Samples are separated into QC batches. This does not include any quality control samples. Open a new QC batch every 20 samples or each time samples are setup.

10.3. Method/Preparation Blank: For every QC batch prepare a preparation blank using all reagents in the same volumes as used in the preparation of samples. Analyze using the same reagents and procedure as other samples. The blank value should fall below the method detection limit. Do not subtract the blank value from sample values.

10.4. Matrix Spike: For every QC batch analyze matrix spike and matrix spike duplicate. The percent recovery should be between 85-115%.

10.4.1. Aqueous spiking procedure:

10.4.1.1. Matrix spike low- Take a sample aliquot in a 50ml volumetric flask and spike with 50ug/L of Cr+6, bring to volume with sample. Add indicator and adjust pH as normal.

10.4.1.2. Matrix spike high- Take a sample aliquot in a 50ml volumetric flask and spike with

200ug/L of Cr+6, bring to volume with sample. Add indicator and adjust pH as normal.

10.4.2. The following calculation should be performed to determine percent recovery.

$$\% \text{ Recovery} \qquad \frac{(SSR - SR)}{SA} \times 100$$

SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

10.5. Post Verification Spike: For every QC batch of a similar matrix, a post verification spike (PVS) will be analyzed. This consists of first, analyzing a sample. Then take a new sample aliquot and spike with two times the concentration of Cr(VI) found in the sample or 15.0 ppb (whichever is greater). Finally add indicator and adjust pH as normal. If necessary, dilute the PVS to fall within the range of the calibration curve.

10.6. Duplicates: For every QC batch of a similar matrix, a duplicate will be prepared exactly the same as the original sample. The relative percent difference(RPD) between the sample and duplicate should be less than or equal to 20%.

10.6.1. The following calculation should be performed to determine RPD.

$$RPD = 2(S-D) \times 100 / (S + D)$$

S = Original Sample Value
D = Duplicate Sample Value

10.7. Laboratory Control Sample (LCS): A laboratory control sample must be analyzed with each group of samples processed. The recovery must be within the vendor-specified limits.

11. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

12. WASTE MANAGEMENT AND POLLUTION PREVENTION

12.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

12.2. POLLUTION PREVENTION:

12.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot

be feasibly reduced at the source, the agency recommends recycling as the next best option.

12.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

13.0 METHOD PERFORMANCE

13.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

13.2 All MDL results are available on file.

14. DEFINITIONS

14.1. Refer to document DEFDOC-04 for definitions.

15. REFERENCES

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

STL Edison Standard Operating Procedure	Revision Date: April 1, 2004
Title: SULFATE, Turbidimetric Analysis of Sulfate in Water and Wastewater and Soil	SOP Number SO4TW04
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Department Manager: _____	
File Location: f:\qaqc\sops\nelac\nelac sops 2004\140wetch\general\so4tw04.doc	

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1. SCOPE AND APPLICATION

1.1. Methods EPA 375.4 and SW846 9038 are described in this SOP for the analysis of sulfate. This SOP is applicable for the analysis of waters and soils.

1.2. The minimum detectable limit is approximately 1.0 mg/L.

2. METHOD SUMMARY

2.1. Sulfate is detected by forming a precipitate of barium sulfate which is suspended. The turbidity formed by this suspension is read by a turbidimeter. Each time samples are analyzed an initial calibration verification, calibration blank and calibration verification standards are analyzed. The results of the standards must be 90-110% recovery. In addition every four samples the ICV/ICB are reanalyzed and must be recovered within the same limits. The blank must be less than the reporting limit. Each batch of samples a laboratory control sample, method blank, matrix spike and matrix spike duplicate are analyzed. The method blank must be less than the reporting limit. The BS, MS and MSD must be recovered within laboratory generated limits and relative percent difference between the MS and MSD must be less than the laboratory generated control limits.

3. APPARATUS AND MATERIALS

3.1. Watch

3.2. Spoon, 5ml

3.3. Magnetic stirrer

3.4. Turbidity meter, Hach 2100A

4. REAGENTS

4.1. Barium chloride, crystals: 20-30 mesh.

4.2. Sodium Chloride

4.3. Hydrochloric Acid

4.4. Isopropanol

4.5. Glycerol

4.6. Conditioning Reagent: Place 75g NaCl in a 500 ml beaker with magnet on magnetic stirrer. Add exactly 300ml deionized water and dissolve completely. Add 30ml conc. HCl and allow to mix. Add 100ml isopropanol (or 95% ethanol) and allow to mix. Finally add 50ml glycerol and allow to mix until solution is homogenous.

5. STANDARDS

5.1. Standard sulfate solution (1000mg/l SO_4): Pre-dry sodium sulfate at 250°C for 2 hours. Dissolve 1.470g in deionized water and dilute to 1000ml.

5.2. Blank spike (500 mg/l SO_4): Pre-dry sodium sulfate (different lot number from the above standard) at 250°C for 2 hours. Dissolve 0.735g in deionized water and dilute to 1000ml.

6. INTERFERENCES

6.1. Silica in concentrations above 500mg/l will interfere with this method.

6.2. Color and turbidity must be accounted for with sample background analysis.

6.3. Sample may be filtered if oily or dirty.

7. SAMPLE HANDLING AND PRESERVATION

7.1. Samples must be maintained at 4°C until the time of analysis.

7.2. The holding time for sulfate is 28 days from the time of sample collection.

7.3. Soils are analyzed by ASTM leachate extraction prior to analysis.

8. SAFETY

8.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

8.3. All questions pertaining to any safety procedure should be brought to the department manager or Edison Safety Officer.

8.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Glycerol is incompatible with strong oxidizers. In the event of a fire the glycerol releases a toxic gas (Acrolein).

8.5. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Barium Chloride	Poison Irritant	0.5 mg/m3 TWA	May be fatal if swallowed. Harmful if inhaled. Avoid contact with eyes, skin, and clothing. Avoid breathing dust. Keep container closed and when in use adequate ventilation.
1 – Always add acid to water to prevent violent reactions.			
2 - Exposure limit refers to the OSHA regulatory exposure limit.			

9. PROCEDURE

9.1. Turn on turbidity meter and set to 100 NTU range. Be sure "riser" is in cell holder. Allow to warm up for at least 1/2 hour (but preferably 1 hour).

9.2. Pour 100ml of sample or ASTM leachate into labeled specimen cup and allow to come to room temperature.

9.3. Prepare calibration standards by adding the amount specified below to 100ml volumetric flasks and bringing to volume with deionized water.

Standard (ml)	Concentration SO_4 (mg/l)
0	0
0.5	5
1.0	10
1.5	15
2.5	25
4.0	40

9.4. Calibrate with 40 PPM standard first, adjusting the meter to read 100 NTU with the "standardize" knob.

9.5. Have clean cell ready to use.

9.6. Place specimen cup on magnetic stirrer. Add magnet and begin stirring.

9.7. Add 5.0 ml conditioning reagent.

9.8. Pour sample into cell and record turbidity as background in workbook.

9.9. Pour contents of cell back into specimen cup with the rest of the sample on stirrer.

9.10. While stirring at constant speed, note time and add a spoonful of barium chloride. Stir EXACTLY one minute.

9.11. Immediately pour sample into cell and place in turbidity meter. If sample falls outside of range of highest standard, dilute another portion of sample with deionized water and reanalyze.

9.12. Record highest value from meter within 4 minutes.

9.13. Rinse cell and proceed with next sample.

9.14. RUN A CCV, CCB EVERY FOURTH SAMPLE.

9.15. Data Processing

9.15.1. Calculations for Water and Soil Samples:

9.15.1.1. Enter workbook data into computer under sulfate spreadsheet.

9.15.1.2. Run linear regression and verify that the correlation coefficient is greater than 0.997.

9.15.1.3. For soil samples, be sure to include 20x dilution factor from the ASTM leachate and % solids.

9.15.2. Results:

9.15.2.1. Transfer results to analyte sheets. Place analyte sheets with spreadsheets in job folders and sign chronicles.

9.15.2.2. Notify supervisor if check standards or quality control samples exceed limits.

9.15.2.3. Submit quality control sheets to manager as required for each batch.

10. QUALITY Control

10.1. Prior to the analysis of any samples the following must be performed.

10.1.1. Method Detection Limit (MDL) -- MDLs must be established by taking seven replicate aliquots of the fortified reagent water and process through the entire analytical method. MDLs should be determined yearly.

10.2. Samples are separated into QA batches. This does not include the blank, duplicate sample and Blank spike. Open a quality control batch every two weeks or every 20 samples, whichever is first.

10.3. For each QA Batch the following procedures must be performed:

10.3.1. A blank must be analyzed each time samples are analyzed. Use deionized water for the blank. The results must be below the reporting limit.

10.3.2. A Blank Spike is performed daily and is used to measure method performance on the matrix being analyzed. The result must recover ± 10% from the true value.

10.3.3. Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked with known amount. The recovery and relative percent difference must be within laboratory generated control limits. Calculate Recoveries and RPD as follows:

$$\%\text{Recovery} = \frac{(\text{MS or MSD result} - \text{sample conc.})}{}$$

x100

Amount spiked

$$RPD = \frac{(ppm\ MS - ppm\ MSD)}{(ppm\ MS + ppm\ MSD)/2} \times 100$$

10.4. Instrument calibration should be verified in the following manner:

10.4.1. After initial calibration, every 4 samples and after last sample, a midpoint standard and blank will be analyzed. Verify ICV/CCV are 90-110% of value and ICB/CCB are below the reporting limit.

11. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated (flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

12. WASTE MANAGEMENT AND POLLUTION PREVENTION

12.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

12.2. POLLUTION PREVENTION:

12.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

12.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

13. METHOD PERFORMANCE

13.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

13.2 All MDL results are available on file.

14. DEFINITIONS

14.1 Refer to document DEFDOC-04 for definitions.

15. REFERENCES

15.1 Methods for Chemical Analysis of Water and Wastewaters, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Method 375.4

15.2 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW846 Method 9038.

STL Edison Standard Operating Procedure	Revision Date: April 1, 2004
Title: NITRATE & NITRITE, Analysis of Nitrate and Nitrite in Water, Wastewater and Soil-Automated Laboratory Director: _____ Technical Director: _____ QA Manager: _____ Department Manager: _____ File Location: f:\qaqc\sops\nelac\nelac sops 2004\140wetch\nitrogen\no32a04.doc	SOP Number NO32A04 Revision 2 Page 1 of 9

1. SCOPE AND APPLICATION

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1.1. Methods EPA 353.2, SM 4500-NO_3F and QuikChem Method 10-107-04-1-C are described in this SOP. These methods are applicable to soils and waters.

1.2. This method can be used to determine nitrate or as a sum of both forms of nitrogen from 0.02-2.0 mg/l and for nitrite from 0.01 - 1.0 mg/L.

2. METHOD SUMMARY

2.1. Nitrate and nitrite are analyzed using an automated reduction and subsequent colorimetric measurement for quantitation. Each batch of twenty samples or less, a laboratory control sample, a matrix spike and matrix spike duplicate are analyzed. The results are compared to laboratory generated control limits. Each time samples are analyzed, an ICV, ICB and method blank are analyzed. The method blank and ICB must be less than the reporting limit. The ICV must recover between 90-110%.

2.2. Nitrate is reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color that is read at 520 nm. Nitrite alone can be determined by removing the cadmium column.

3. INTERFERENCES

3.1. Filter samples if sample has particulates, color, or is turbid. Use a 0.45 um filter syringe.

4. APPARATUS AND MATERIALS

4.1. Analytical balance, capable of accurately weighing to the nearest 0.0001g.

4.2. Cd-Cu Reduction Column.

4.3. pH meter.

4.4. Two state switching valve.

4.5. Class A volumetric flasks and pipettors- various

4.6. Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

4.7. Autosampler

4.8. Multichannel proportioning pump

4.9. reaction unit and manifold

4.10. Colorimetric detector, 540nm.

4.11. Data system

5. REAGENTS

5.1. 15 N NaOH: Slowly add 150g to 250 ml DEIONIZED WATER. CAUTION: The solution is very hot! Cool and stored in plastic bottle. (Stable for 6 months).

5.2. Ammonium Chloride buffer, pH 8.5: In a 1L beaker, dissolve 85.0g ammonium chloride (Aldrich-NH_4Cl) and 1.0g disodium tetraacetic acid dihydrate ($Na_2EDTA*2H_2O$) in about 800ml DEIONIZED WATER. Dilute to mark and invert to mix. Adjust the pH to 8.5 with 15N NaOH. (Stable for 6 months).

5.3. Sulfanilamide color reagent: In a 1 L beaker containing about 600 ml of DEIONIZED WATER, add 100 ml of 85% phosphoric acid (H_3PO_4), 40.0 g sulfanilamide, and 1.0 g N-(1-naphthyl)ethylenediamine dihydrochloride (NED). Stir with stir

bar for about 30 minutes until dissolved. Transfer to a 1L volumetric flask, dilute to mark and mix. (Stable for 1 month).

6. STANDARDS

6.1. Stock Nitrate standard: 2000 mg N/L as NO_3-: In a 1 L volumetric flask, dissolve 14.44 g potassium nitrate (KNO_3) in about 600 ml DEIONIZED WATER. Add 2 ml of chloroform. Dilute to mark and invert to mix. Refrigerate. This solution is stable for 6 months.

6.2. Stock Nitrite Standard, 2000 mg N/L as NO_2-: In a 1 L volumetric flask, dissolve 9.86 g sodium nitrite ($NaNO_2$) or 12.14 g potassium nitrite (KNO_2) in approximately 800 ml DEIONIZED WATER. Add 2 ml of chloroform. Dilute to mark and invert to mix. Refrigerate. Stable for 6 months.

6.3. Mixed Nitrate/Nitrite working solution, 20.0 mg N/L as NO_3- and NO_2-: In a 100 ml volumetric flask, pipet 500 ul of Nitrate Stock and 500 ul of Nitrite stock. Dilute to mark and mix. Prepare weekly.

6.4. Prepare Nitrate/Nitrite standard using Nitrate/Nitrite working solution in 100 ml of water.

Standard Sol'n (ml)	Concentration of NO_3 (ppm)	Concentration of NO_2 (ppm)
0	0	0
0.25	0.05	0.025
0.50	0.10	0.050
2.5	0.50	0.25
5.0	1.0	0.50
7.5	1.5	0.75
10.0	2.0	1.0

7. PRESERVATION AND HANDLING

7.1. Nitrite determinations must be performed on an unpreserved sample refrigerated to 4°C. The holding time is 48 hours.

7.2. Nitrate determinations must be performed on an unpreserved sample refrigerated to 4°C. The holding time is 48 hours.

7.3. Nitrate plus Nitrite can be performed on a sample preserved to pH<2 with Sulfuric Acid and refrigerated to 4°C. The holding time is 28 days.

8. SAFETY

8.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

8.3. All questions pertaining to any safety procedure should be brought to the department manager or Edison Safety Officer.

8.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

8.5. PRIMARY MATERIALS USED

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

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Ammonium Hydroxide	Corrosive Poison	50 ppm-TWA	Vapors and mists cause irritation to the respiratory tract. Causes irritation and burns to the skin and eyes.
Chloroform	Carcinogen Irritant	50 ppm Ceiling	Acts as a relatively potent anesthetic. Irritates respiratory tract and causes central nervous system effects, including headache, drowsiness, dizziness. Causes skin irritation resulting in redness and pain. Removes natural oils. May be absorbed through skin. Vapors cause pain and irritation to eyes. Splashes may cause severe irritation and possible eye damage.
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Potassium Nitrate	Oxidizer	None	Causes irritation to the respiratory tract, skin and eyes. Symptoms may include coughing, shortness of breath. Symptoms include redness, itching, and pain.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

9. PROCEDURE

9.1. Use NaOH or H_2SO_4 to adjust pH = 5 -9.

9.2. Follow manifold scheme of this method. Place all reagent lines in DEIONIZED WATER to check for leaks and flow rate. Turn pump on.

9.3. Switch reagent lines into proper reagent for approximately 5 minutes before putting the column on line.

9.4. Set up tray table with all appropriate QA checks (includes sample, matrix spike, matrix spike duplicate, and LCS. Run a CCV/CCB every 10 samples and at the end of the run. CCV must have a % recovery of 90-110%.

9.5. Place standards and samples into the autosampler cups. The following analytical sequence must be used:

Calibration
AICV/ACCV
ICB
10 samples
ACCV1

CCB1
Repeat until run is complete
ACCV2
CCB2

9.6. The column should be on line for approximately 15 minutes before running the tray. This allows the baseline to stabilize.

9.7. Run tray.

9.8. Make all appropriate dilutions, if necessary.

9.9. Correlation coefficient of 0.997 or greater must be obtained.

9.10. Data Processing:

9.10.1. Record results in workbook and transfer to analyte sheet.

9.10.2. Place analyte sheets in job folders and sign chronicles.

9.10.3. Notify supervisor if quality control limits are exceeded.

9.10.4. Submit quality control sheets to manager as required for each batch.

10. QUALITY CONTROL

10.1. Prior to the analysis of any samples the following must be performed.

10.1.1. Method Detection Limit (MDL) -- MDLs must be established by taking seven replicate aliquots of the fortified reagent water and process through the entire analytical method. MDLs should be determined yearly.

10.2. Samples are separated into QA batches. This does not include the blank, duplicate sample and Laboratory Control Sample. Open a quality control batch every two weeks or every 20 samples, whichever is first.

10.3. For each QA Batch the following procedures must be performed:

10.3.1. A blank must be analyzed each time samples are analyzed. Use deionized water for the blank. The results must be below the reporting limit.

10.3.2. A Laboratory Control Sample is obtained from an outside vendor and is used to measure method performance on the matrix being analyzed.

10.3.3. Two portions of the same sample (matrix spike and matrix spike duplicate) are spiked with **100 ug** of nitrate. The recovery and relative percent difference must be within laboratory generated control limits. Calculate Recoveries and RPD as follows:

$$\% \text{ Recovery} = \frac{\text{MS(or MSD) result} - \text{sx conc.} \times 100}{\text{Amount spiked}}$$

$$\text{RPD} = \frac{2 \times (\text{ppm MS} - \text{ppm MSD}) \times 100}{(\text{ppm MS} + \text{ppm MSD})}$$

10.4. Instrument calibration should be verified in the following manner:

10.4.1. The standard curve must have a correlation coefficient no less than or equal to 0.997.

10.4.2. After initial calibration, every 10 samples and after last sample, a midpoint standard and blank will be analyzed. Verify ICV/CCV are 90-110% of value and ICB/CCB are below the reporting limit.

11. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

12. WASTE MANAGEMENT AND POLLUTION PREVENTION

12.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

12.2. POLLUTION PREVENTION:

12.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

12.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

13.0 METHOD PERFORMANCE

13.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

13.2 All MDL results are available on file.

14. DEFINITIONS

14.1. Refer to document DEFDOC-04 for definitions.

15. REFERENCES

15.1. Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 4500 NO_3 F.

15.2. Methods for Chemical Analysis of Water and Wastewaters, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Method 353.2.

15.3. EPA Test Method -353.2, Standard Methods, 18th edition, Test Method- 4500-NO3 F, QuikChem Method 10-107-04-1-C.

STL Edison Standard Operating Procedure	Revision Date: April 1, 2004
Title: BIOLOGICAL OXYGEN DEMAND, Analysis of Biological Oxygen Demand in Water and Wastewater	SOP Number Bod04
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1. SCOPE AND APPLICATION

1.1. Methods EPA 405.1 and SM 5210B are applicable to municipal and industrial wastes for the analysis of BOD.

1.2. This method is sensitive to approximately 5 mg/l.

2. METHOD SUMMARY

2.1. Biochemical oxygen demand (BOD) measures the amount of oxygen required for bacteria and other microorganisms to metabolize organic and some inorganic waste material in a water stream. Each time samples are performed the method blank must have an oxygen depletion of less than 0.2 mg/L. Each batch of 20 samples or less a matrix spike, matrix spike duplicate and Laboratory Control Sample are performed. The results are compared to laboratory generated control limits.

2.2. A sample aliquot is diluted with an air saturated deionized water that contains nutrients and seeded with microorganisms. The DO is recorded before and after a 5 day incubation period. The BOD is determined by the difference in DOi and DOf.

2.3. This method is sensitive to approximately 5 mg/l.

3. INTERFERENCES

3.1. All glassware should be thoroughly rinsed with deionized water prior to beginning any procedure.

3.2. If using an industrial seed make sure the seed is viable enough to uptake adequate amounts of DO.

3.3. Discard any reagents if there is any sign of biological growth.

4. APPARATUS AND MATERIALS

4.1. Glass incubation bottles with a 300 ml capacity , glass stopper and plastic cap.

4.2. Aeration pump and tubing.

4.3. Class A volumetric flasks-various

4.4. Class A volumetric pipets-various

4.5. Graduated cylinders-various

4.6. Graduated pipets-various

4.7. Incubator that maintains 20°C

4.8. Dissolved oxygen meter

4.9. Class A burette

4.10. Erlenmeyer flasks

4.11. Five gallon carboy

4.12. Beakers

5. REAGENTS

5.1. Prepare reagents as stated below and reprepare if any sign of biological growth appears.

5.2. Phosphate buffer solution: Dissolve 8.5g KH_2PO_4, 21.75g K_2HPO_4, 33.4g $NaHPO_4 \times 7H_2O$ and 1.7g NH_4Cl in about 500 ml deionized water and dilute to 1 liter. The pH should be 7.2 without further adjustment.

5.3. Magnesium sulfate solution: Dissolve 22.5g $MgSO_4 \times 7H_2O$ in one liter of deionized water.

5.4. Calcium chloride solution: Dissolve 27.5g $CaCl_2$ in one liter of deionized water.

5.5. Ferric chloride solution: Dissolve 0.25g $FeCl_3$ x $6H_2O$ in one liter of deionized water.

5.6. Sulfuric acid (1N): Dilute 28 ml concentrated H_2SO_4 to one liter with deionized water.

5.7. Sodium hydroxide(1N): Dissolve 40g NaOH in one liter of deionized water.

5.8. Sodium sulfite solution (0.025N): Dissolve 1.575g Na_2SO_3 in one liter of deionized water. This solution is unstable; prepare daily.

5.9. Potassium iodide paper

5.10. Seed (Polyseed or an industrial source)

5.11. Nitrification Inhibitor- Formula 2533 purchased from Hach Inc.

5.12. BOD Nutrient buffer pillows: purchased commercially.

6. STANDARDS

6.1 Laboratory Control Sample (LCS): purchased commercially .

7. PRESERVATION AND HANDLING

7.1. The holding time for BOD in water is 48 hours from sample collection.

7.2. Samples must be collected and stored at 4 degrees Celsius with no preservatives added.

8. SAFETY

8.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

8.2. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

8.3. All questions pertaining to any safety procedure should be brought to the department manager or Edison Safety Officer.

8.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

There are no specialized safety concerns associated with this method.

8.5. PRIMARY MATERIALS USED

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

9. PROCEDURE

9.1. Nutrient (or Dilution) Water can be prepared by one of the following methods - 8.1.1 or 8.1.2.

9.1.1. Fill carboy with 6 gallons or 24 liters of deionized water. Add 24 ml of each of the following:

9.1.1.1. Phosphate buffer solution

9.1.1.2. Ferric chloride solution

9.1.1.3. Calcium chloride solution

9.1.1.4. Magnesium sulfate solution

8.1.2 Fill carboy with 6 gallons or 24 liters of deionized water and add 4 of the BOD Nutrient buffer pillows. (1 pillow per 6 liter of DI water).

8.1.3 Aerate the carboy for a minimum of 2 1/2 hours prior to the start of setting samples. This should be sufficient time to saturate the nutrient water with oxygen to achieve a DO reading of 7.0-9.0 mg/l.

9.2. Seed the Dilution Water:

9.2.1. A commercial or natural source from an industrial effluent may be used as seed for this test. Use one of the following methods to prepare seed for the nutrient water.

9.2.2. Prepare the Polyseed:

9.2.2.1. Place one polyseed capsule in 500 ml deionized water and stir for one hour. Prepare daily.

9.2.3. Preparation of Natural Seed

9.2.3.1. Fill a 2 gallon plastic container with activated sludge from MCUA and keep aerated.

9.2.3.2. Feed daily by discarding approximately 500 ml of the activated sludge and replenishing with approximately 500 ml of influent from MCUA.

9.2.3.3. While aerating:

9.2.3.3.1. Take 500 ml aliquot from the activated sludge.

9.2.3.3.2. Let it sit for 30 minutes.

9.2.3.3.3. Decant upper liquid and use for seeding.

9.3. Calibrate the Dissolved Oxygen Meter:

9.3.1. Aerate enough deionized water to fill 2 BOD bottles.

9.3.2. Measure the DO reading of one bottle with the DO probe.

9.3.3. Perform the Winkler titration as per Edison SOP DOWIN01 on the second bottle to determine DO. Use 200 ml instead of 300 ml.

9.3.4. Record the DO readings from the Winkler titration in the BOD logbook.

9.3.5. Adjust the DO meter to the DO measured from the Winkler titration.

9.4. Sample Pretreatment:

9.4.1. Measure the pH of each sample . If the pH is not within 6.5 - 7.5 take a sample volume sufficient enough to set 3-4 dilutions' and adjust accordingly with the sulfuric acid solution or sodium hydroxide solution. Do not change volume more than 0.5%.

9.4.2. Measure the presence of chlorine residual in each sample by placing a drop or two of sample on a strip of KI paper. If the presence of chlorine residual is detected, analyze a sample aliquot with the DPD method. Add to another sample aliquot (sufficient enough to set 3-4 dilutions) the relative volume of Na_2SO_3 solution determined by the DPD titration.

Note: Excess Na_2SO_3 will exert an oxygen demand.

9.5. Procedure for Biochemical Oxygen Demand:

9.5.1. Fill each BOD bottle approximately half with the nutrient water

9.5.2. Measure 3-4 varying sample volumes for each sample and place in a BOD bottle. The aliquots should be such to uptake 2.0 mg/l of DO but not more than 90% of the initial DO. (Example of sample volumes 5ml, 10ml, 25ml, 50ml or 50ml, 100ml, 200ml.) The maximum sample aliquot is 200 ml. If a sample aliquot of 0.5 ml or less is required, dilute by serial dilution.

Note: Refer to the historical data of the sample for proper dilutions. A quick way to estimate the BOD concentration is to determine the COD and divide by two.

9.5.3. Add 3ml of the seed solution to each BOD bottle (except the preparation blank and seed control.)

9.5.4. Fill each BOD bottle with aerated water. Make sure there are no air bubbles in the bottles. If so, gently tap the outside of the BOD bottle with the glass stopper until no air bubbles are present.

9.5.5. Measure the initial DO (DOi) with the calibrated meter on all sample dilutions. Record the DOi in the BOD logbook.

9.5.6. Place a ground glass stopper in each bottle and cover with a plastic cap.

9.5.7. Place in dark incubator for 5 days (BOD5) or 20 days (BOD20) at 20C $\pm$1 °C.

9.5.8. Remove BOD bottles at the end of the incubation period and record the final DO (Dof) with a calibrated DO meter. Record DOf in the BOD logbook.

9.6. Procedure for Carbonaceous Biochemical Oxygen Demand

9.6.1. Follow the same procedure as above with the exception of adding 0.16 g of Nitrification Inhibitor Formula 2533 or 2 shots to each BOD bottle.

9.7. Data Processing:

9.7.1. Calculation:

$$BODmg/l = \frac{(A - B) - C \times 300 \times D}{E}$$

A = Initial dissolved oxygen reading
B = Final dissolved oxygen reading
C = Seed correction factor. See Section 10.3.2
D = Dilution factor
E = Sample volume

10. QUALITY CONTROL

10.1. Prior to the analysis of any samples the following must be performed.

10.1.1. Method Detection Limit (MDL) -- MDLs must be established by taking seven replicate aliquots of the fortified reagent water and process through the entire analytical method. MDLs should be determined yearly.

10.2. Samples are separated into QA batches. This does not include the blank, MS, MSD, and LCS. Open a quality control batch every two weeks or every 20 samples, whichever is first.

10.3. For each QA Batch the following procedures must be performed:

10.3.1. A blank must be analyzed each time samples are analyzed. Oxygen depletion should be no more than 0.2mg/L.

10.3.2. Five raw seed control samples are set each time. Withdraw 100 ml of the raw seed into a 1000 ml volumetric flask. Dilute to the mark with deionized water and transfer to a 1000 ml beaker. Add 5,10, 20, 30 and 50 ml of the diluted seed to BOD bottles and fill to the top with dilution water. The raw seed control samples monitor the viability of the seed. Use the results of the one that has a DO uptake between 0.6-1.0 mg/L to calculate the seed correction factor.

10.3.3. An LCS must also be run for each time. The recovery must be within manufacturer's control limits.

10.3.4. MS and MSD are prepared by setting two sets of different dilutions of the sample and spiked with the LCS. The recovery and relative percent difference must be within manufacturer's control limits.

$$\text{Recovery} = \frac{(\text{MS or MSD Conc.} - \text{sample conc.})}{\text{Amount spiked}} \times 100$$

$$\text{RPD} = 2 \times \frac{(\text{ppm MS} - \text{ppm MSD})}{(\text{ppm MS} + \text{ppm MSD})} \times 100$$

11. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated (flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

12. WASTE MANAGEMENT AND POLLUTION PREVENTION

12.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

12.2. POLLUTION PREVENTION:

12.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

12.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

12.3 Waste Streams Produced by the Method

There are no special wastes produced by this method.

13. METHOD PERFORMANCE

13.1 A method detection Limit (MDL) study is performed annually for this analysis. The MDL study is conducted by taking seven replicate preparations of a low-concentration standard (approximately 2-5 times the estimated method detection limit) through all sample preparation steps and analysis for the specified analytical method. MDL values are calculated from the standard deviation of analytical results and the Students' t value for the number of replicate samples analyzed. The resulting calculated MDL values are evaluated by the Laboratory Manager and the QA Manager against criteria to determine their acceptability.

13.2 All MDL results are available on file.

14. DEFINITIONS

14.1 Refer to document DEFDOC-04 for definitions.

15. REFERENCES

15.1 Methods for Chemical Analysis of Water and Wastes, EMSL-Cincinnati, EPA/600/4-79-020, March 1983 and 1979; Test Method 405.1.

15.2 Standard Methods for the Examination of Water and Wastewater , 18th Edition, American Public Health Association, Baltimore Maryland, 1992, SM 5210.

STL-Edison Standard Operating Procedure	Revision Date: March 9, 2004
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1. SCOPE and APPLICATION

1.1. The following procedure outlines the steps taken to ensure that instruments and ancillary equipment are in condition to perform their respective functions.

2. PROCEDURE

2.1. Analytical Instruments - The maintenance procedures, calibration procedures and tuning procedures which are carried out by analysts are covered in detail in the analytical SOPs. The department manager is responsible for the maintenance of the instruments within his laboratory. Where applicable, analytical instruments are covered by a service contract. All information related to the calibration and maintenance of analytical instruments will be recorded in an individual laboratory notebook.

2.2. Ancillary Equipment - The inorganic laboratory manager is responsible for all the ancillary equipment listed below except for the GC items which are the responsibility of the GC manager and refrigerators which are the responsibility of the login group. In addition to routine instrument maintenance provided by manufacturer's maintenance contracts and software services, STL-Edison will perform the following checks to insure that ancillary equipment and instrumentation are capable of functioning properly:

2.2.1. Analytical Balances

2.2.1.1. The balance is to be certified and checked once a year by a balance servicing company.

2.2.1.2. The analytical balance is to be checked once per day with class S weights, over the working range of the balance.

2.2.1.3. All pertinent information will be recorded in a bound logbook.

2.2.2. pH Meters

2.2.2.1. Meters are to be standardized against two buffers that bracket the pH of the sample.

2.2.2.2. The electrodes will be immersed in an appropriate buffer or water when not in use, and filled with an appropriate filling solution specified by the manufacturer.

2.2.2.3. pH meters are calibrated daily by using buffers 4,7 and 10. The actual readings, date, time and analyst's initials are recorded in a bound logbook.

2.2.3. Spectrophotometers

2.2.3.1. A monthly wavelength calibration of Spectrophotometers will be performed for various general chemistry determinations.

2.2.3.2. The wavelength observed, date of check and analyst's name will be recorded in a bound logbook.

2.2.4. Drying Ovens

2.2.4.1. The temperature of each drying oven will be recorded daily in a bound logbook.

2.2.5. Refrigerators

2.2.5.1. The computerized temperature maintenance program prints the temperature of each refrigerator on a daily basis. This program records the temperature of each refrigerator every 15 minutes. If the temperature is out of the specified range for more than 3 hours, the login supervisor notifies the QA manager and the affected department manager. The Health and Safety coordinator is also notified for any maintenance or repair needed. All corrective action performed is documented in the refrigerator maintenance logbook.

2.2.6. Thermometers

2.2.6.1. All glass thermometers will be verified yearly by comparing the readings of those thermometers with a NBS traceable certified thermometer. Non-glass thermometers will be verified on a quarterly basis. Each thermometer will be identified and a record will be maintained including thermometer identification, the temperature of the certified thermometer, the temperature of the thermometer being verified, date of verification and analyst who performed verification.

2.2.6.2. The NBS thermometer is calibrated every three years by a certified outside contractor. The recertified thermometer is returned with a certificate of accuracy and is retained on file in the Wet Chemistry Manager's office.

2.2.7. GC, GC/MS Detectors

2.2.7.1. A record will be maintained for each detector with the serial number, date of installation, and background current profiles obtained at the time of installation.

2.2.8. Gas Chromatograph Columns

2.2.8.1. A maintenance logbook is kept for each instrument. This documents the column ID/serial number, part number, date of packing or purchase and the date installed. Packing slips are filed with the Restek Consignment Program kept in the Operations manager's office.

2.2.9 GPC (Gel Permeation) System
On a daily basis, when the system is idle, the following items are checked on each GPC system:

2.2.9.1 The solvent reservoirs are filled with Methylene Chloride to prevent drying of columns.
2.2.9.2 The UV detector is turned off to prevent burning out of lamp.
2.2.9.3 The flow rate on each pump is set at 1 ml/min.

2.2.10 Whenever the GPC is going to be used to cleanup extracts, the following items are checked prior to initiation of the analytical run:

2.2.10.1 The auto sampler is to be purged of air by using the appropriate software system.
2.2.10.2 The UV detector is turned on 30 minutes prior to initiation of the run.
2.2.10.3 The abundance of the UV detector should be zeroed after the warm up period and the desired flow is set.
2.2.10.4 The flow rate on the pump is set at 5 ml/min.
2.2.10.5 The pressure reading on the pump is checked and recorded in the GPC injection log. Any change in the pressure may indicate a leak or block.

2.2.11 Water baths and Incubators

2.2.11.1 Water baths and incubator temperatures are checked twice daily at least 4 hours apart on each day of use. The temperature of each water bath and incubator, date and time of check and the analyst performing the checks are recorded on spreadsheets, which are bound at the end of the year. The bound spreadsheets are given a STL logbook number.

2.2.12 Autoclave

2.2.12.1 Autoclave shall initially demonstrate that it can meet its specified temperature tolerances. Microbiology department shall run different checks as scheduled and shall record all checks on the Autoclave logbook.

For every cycle:

a) Record autoclave operations including the date, contents, maximum temperature reached, pressure time in sterilization mode, total run time (time in and time out), and the analyst's initials.
b) A temperature sensitive tape shall be used with the contents of each autoclave run to show that the autoclave has reach its temperature.

Monthly:

Determine effective sterilization by using biological indicators.

Quarterly:

The autoclave's timing devise is checked against a stopwatch and

checks are documented in the Autoclave bound logbook.

Annually:

Pressure check and temperature device are calibrated on an annual basis by an outside contractor. Records are maintained in the microbiology laboratory.

2.2.13. Ambient temperature of Microbiology room

2.2.13.1 The ambient temperature of the microbiology laboratory is recorded daily in a bound notebook by reading an ambient temperature thermometer mounted on the wall.

3. SAFETY

3.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

3.2. Eye protection, lab coat and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled.

3.3. All questions pertaining to any safety procedure should be brought to the department manager or Edison Safety Officer.

3.4. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None

3.5. PRIMARY MATERIALS USED

There are no materials used in this method that have a serious or significant hazard rating. A complete list of materials used in the method can be found in the reagents and materials section.

Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

4.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

4.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

4.2. POLLUTION PREVENTION:

4.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

4.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

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1. SCOPE AND APPLICATION

The purpose of this SOP is to ensure the quality system is reviewed at least once per year for suitability and effectiveness and to introduce any necessary changes or improvements in the quality system or laboratory operations.

2. METHOD SUMMARY

2.1 This Standard Operating Procedure (SOP) describes the laboratory's quality system management review process. Management continuously reviews the Quality System to ensure its continuing suitability, effectiveness and to introduce any necessary changes or improvements. This procedure describes the processes and documentation of these reviews.

2.2 This SOP applies to the monthly reports to management generated by the QA department and other departments, annual Quality Assurance Plan review and/or revision by the QA department, and weekly staff meetings.

2.2.1 The Monthly Report to Management includes:

2.2.1.1 Client complaints and compliments supplied by Project Management department

2.2.1.2 The number of samples analyzed outside holding time supplied by the department managers and the project management department.

2.2.1.3 The QA department supplies all other parameters in the monthly report. This includes but is not limited to proficiency testing (PT) sample results, audit performance, and certification additions/losses.

2.2.1.4 Turnaround time (TAT) supplied by the analytical and Project Management department etc.

3. RESPONSIBILITIES:

3.1 It is the responsibility of the Laboratory Director to ensure STL Edison personnel document and initiate corrective action for out-of-control events, client complaints, failure to meet client or regulatory requirements, audit findings, PT failures, etc.

3.2 It is the responsibility of the Laboratory Director and QA Manager to adhere to the guidelines and procedures outlined in this SOP.

3.3 It is the responsibility of the Laboratory Director and the QA Manager to ensure that this SOP actually reflects what is being performed in the laboratory.

4. INTERFERENCES:

N/A

5. APPARATUS AND MATERIALS

N/A

6. REAGENTS

N/A

7. STANDARDS

N/A

8. SAMPLE COLLECTION, PRESERVATION AND HANDLING

N/A

9. SAFETY

N/A

10. CALIBRATION & STANDARDIZATION

N/A

11. PROCEDURE

11.1 Monthly Reports to Management

11.1.1 Monthly quality reports are distributed to the Corporate Quality Assurance Director, the Laboratory Director, and the Laboratory Department managers. These reports contain assessments and measurements on the effectiveness of the quality system.

11.1.2 Information from the monthly quality reports can be helpful in establishing preventive actions and improvement goals. The QA monthly reports communicate quality status and needs to upper management. By providing summaries of quality data and analysis of relevant situations, these reports provide management a feedback loop to monitor the effectiveness of laboratory improvement activities.

11.1.3 QA reports distinguish between major and minor issues, while minimizing false alarms. The reports emphasize objective quality measurements, but when subjective elements are reviewed, these are reported without blame.

11.1.4 Examples of items included in monthly quality reports are listed below:

11.1.4.1 Monthly quality reports prepared by the QA department

- Internal and external audit report summaries
- Proficiency Testing (PT) grades
- Project Management report for details of client complaints, compliments and resolutions to complaints/problems/issues
- Summaries of final and raw data review by the QA department
- Holding time issues

11.2 Quality Assurance Manual Review

11.2.1 The QA department reviews the STL Edison Laboratory QAM (Quality Assurance Manual) on an semi annual or annual basis and makes revisions as necessary. The STL Edison QAM must comply with NELAC.

11.3 Weekly Status Meetings

11.3.1 On an as-needed basis, the QA department sends out e-mail summarizing all items due to the QA department, resulting from audits, proficiency testing samples, data review, document control, training, etc.

11.3.2 The department manager must meet internal deadlines resulting from STL Edison quality systems such as annual review of SOPs, SOP training, internal audit responses, etc. The department manager must also meet external deadlines set by auditing agencies, regulatory agencies, and clients. If a department manager cannot meet a deadline, they must notify QA immediately and agree on a new deadline. It is recommended that the department manager set realistic deadlines.

11.4 Annual Management Review

11.4.1 An annual review of the quality system is completed by the Laboratory Director to evaluate its continuing suitability and effectiveness and make any necessary changes or improvements.

11.4.2 The annual management review is documented in memo form from the Laboratory Director to the Quality Assurance Manager.

11.4.3 The management review shall be conducted at least annually and include:

- Suitability of policies and procedures
- Management reports
- Summarizing internal audit results
- Corrective and preventive actions
- Assessments by accrediting bodies or regulatory agencies
- Results of proficiency tests
- Changes in the volume or type of testing
- Client complaints and communications
- Staffing resources and training requirements

12. CALCULATIONS

N/A

13. QUALITY CONTROL

N/A

14. DATA ASSESSMENT AND ACCEPTANCE CRITERIA FOR QUALITY CONTROL MEASURE

N/A

15. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA. Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

16. WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all

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

16.2. POLLUTION PREVENTION:

16.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

16.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

17. METHOD PERFORMANCE

N/A

18. DEFINITIONS:

Refer to document DEFDOC-04 for definitions.

1. SCOPE and APPLICATION

1.1. This SOP outlines the steps taken to eliminate data entry errors and to maintain the security of the databases and data systems.

1.2. The mechanism for tracking sections of data packages is also discussed.

2. PROCEDURE

2.1. CONTROLLING AND ESTIMATING DATA ENTRY ERRORS

2.1.1. The data reporting system is designed to input analytical results directly from analytical instruments via a network. This minimizes the number of transcriptions, which in turn minimizes the potential for a reporting error.

2.1.2. Reviewed, processed data is uploaded from instruments to the appropriate reporting database.

2.1.3. The department manager or supervisor checks manually entered data. Any errors found are reviewed and compared with the raw data before re-updating in the LIMS.

2.2. REVIEWING CHANGES TO DATA AND DELIVERABLES AND ENSURING TRACEABILITY OF UPDATES

2.2.1. All applicable data to be used in the production of the data report is kept in the Report production area.

2.2.2. Data repositories such as instrument run logs, extraction logbooks and maintenance logs are kept in bound, paginated books. The signature of the responsible analyst is subjected to verification by the respective supervisor. Logbooks that become

filled are stored in a secured cabinet in the laboratory and periodically archived for storage.

2.2.3. As data is produced, it may be tracked through an individual department by means of the information contained in the job folder and the run logs or extraction logs maintained in the individual department.

2.2.4. The reporting databases are secured by means of a password protection system that accounts for any changes that are made to the database. An audit trail is created to reconstruct what changes were made and who made them.

2.2.5. The job folder acts as the repository for all data pertaining to the specific group of samples. After the report is sent out, the Report Production Department personnel archive the job folder in increasing job number order. The job folders are kept on site for a period of not less than five years and are easily accessible if information is requested at a later date.

2.3. TESTING, MODIFYING, AND IMPLEMENTING CHANGES TO EXISTING COMPUTER SYSTEMS INCLUDING HARDWARE, SOFTWARE, AND DOCUMENTATION OR INSTALLING NEW SYSTEMS.

2.3.1. All modifications to computer systems or new system installations are coordinated by the MIS Manager.

2.3.2. Initially, a meeting is held with but not limited to the MIS Manager, Lab Manager, Quality Assurance Manager, and pertinent department managers. System enhancements and development are discussed and organized at this meeting. Upon satisfactory agreement of all parties, the scope of the development is defined.

2.3.3. Testing is performed throughout the development cycle by the development staff. Depending on the scope of the System Development Project, lab personnel will be involved with testing either throughout the development cycle or at the end for final testing and approval.

2.3.4. Where possible, any new or modified systems are tested side-by-side with existing systems to ensure accuracy and to limit the introduction of new and unforeseen '*bugs*.'

2.3.5. Documentation is maintained during the development cycle for system administration and maintenance purposes. End-user documentation is created at the end of the cycle to be included with any end-user training needed. In addition, any SOPs that are affected by these changes are modified.

2.3.6. Finally, upon approval of the Lab Manager and the Quality Assurance Manager, the new development or modifications are implemented and become standard operating procedures.

2.4. DATABASE SECURITY, BACKUP AND ARCHIVAL

2.4.1. All electronic data is archived from analytical instruments and data reporting databases using a variety of media ranging from Digital Data Storage (DDS) tapes for GC and GC/MS data to cassette tapes and floppy disks for metals data. Archived data is indexed and cross-referenced to instrument run logs to facilitate retrieval if necessary.

2.4.2. All electronic data is archived in duplicate. One copy is maintained at the main facility for quick retrieval and the other copy is maintained at a remote location for disaster recovery.

2.4.3. Database security is maintained by limiting access rights through password protection. In general, users are granted the minimum amount of privilege needed to perform their respective job functions.

2.4.4. Audit Trails are maintained on all databases to monitor data manipulation and modifications.

2.5. SYSTEM MAINTENANCE

2.5.1. All routine maintenance procedures are documented in a manual that is maintained by the MIS Manager. This manual contains step-by-step procedures for administering critical system activities including; backups, retrievals, user maintenance, and security procedures.

2.5.1.1. Hardware

2.5.1.1.1. Data systems are continually monitored by the MIS Manager to ensure proper operation and full functionality.

2.5.1.1.2. Where critical, redundancies are built-in to the system to maintain full system operation in the event of a critical hardware failure. These redundancies usually involve maintaining a backup system, which can replace a main system until that main system is repaired or replaced.

2.5.1.1.3. Where possible and cost-effective, replacement hardware is stockpiled for emergency.

2.5.1.1.4. Backup systems are routinely tested, specifically when normal system maintenance requires the shutdown of the main systems.

2.5.1.1.5. Maintenance contracts are maintained with vendors for all critical hardware.

2.5.1.1.6. Response times for in-house maintenance are not more than six hours. Response times for maintenance contracts are not more than 24 hours.

2.5.1.1.7. All systems are protected against electronic surges or spikes and critical systems are protected by Uninterruptible Power Supplies in the event of power failures.

2.5.1.2.Software

2.5.1.2.1.Telephone support contracts are maintained with vendors for all critical systems software. Response time for critical problems is not more than 4 hours.

2.5.1.2.2.A close working relationship is maintained with all critical software vendors to ensure software compliance with all methods and certifications.

2.5.1.2.3.Custom software is developed and maintained by the in-house staff, where a suitable third-party package can not be found.

2.5.1.2.4.All electronic media which enters or leaves the lab, is checked against the latest anti-virus software packages.

2.6. SYSTEM MANAGEMENT RESPONSIBILITY

2.6.1. MANAGEMENT INFORMATION SYSTEM MANAGER

2.6.1.1. The maintenance of all system hardware including but not limited to; computers, network hardware, printers, and other peripherals.

2.6.1.2. Ensuring the proper operation, installation, and availability of all software such as: database management systems, data reporting, data acquisition, general office packages, operating systems, and network operations.

2.6.1.3. The operation and availability of the computer network.

2.6.1.4. Data backup, archival, and retrieval.

2.6.1.5. System and Database Security.

2.6.1.6. Evaluation, acquisition, and implementation of new systems and software.

2.6.1.7. All in-house software development.

2.6.1.8. Technical and Software Support.

2.6.1.9. General end-user training.

2.6.1.10. Software compliance with existing Certifications and Contracts

2.6.1.11. Data Integrity

2.6.1.12. Data Systems Operations

2.6.1.13. Data System Support and Training.

2.7. STAFF TRAINING PROCEDURES

2.7.1. Staff training in data systems is the responsibility of the respective department manager. Where such media exists, training videos or multimedia presentations are used to introduce the trainee to the data system or software application. The user is then given any SOP or software documentation available as additional introduction to the system.

2.7.2. After initial exposure, a system expert will provide one-on-one support to the trainee detailing the specific operation of the package and how the system is to be used to perform the job at hand.

2.7.3. Upon satisfactory completion of the training, the trainee's manager will determine if the user is ready to work or if more training is needed.

2.7.4. When the new user's training is approved, a password and security privileges are assigned and the new user is allowed to work with the system.

3. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

4. WASTE MANAGEMENT AND POLLUTION PREVENTION

4.1. WASTE MANAGEMENT:

The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

4.2. POLLUTION PREVENTION:

4.2.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes

cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

4.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

5. DEFINITIONS

5.1. Refer to document DEFDOC-04 for definitions.

6. REFERENCES

6.1. EPA CLP SOW OLM04.3.

6.2. CLP SOW ILM0 5.2.

6.3. EPA Test methods for Evaluating Solid Waste, SW846 Third Edition including revisions.

STL Edison Standard Operating Procedure	Revision Date: 03/09/2004
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1. SCOPE and APPLICATION

1.1. This procedure addresses the mechanism for centralizing the documents supporting a sample submission and for retrieval of these documents.

1.2. This procedure also addresses the maintenance of all notebooks used in the laboratory.

2. PROCEDURE

2.1. Supporting documents per sample submission

2.1.1. A group of samples submitted for analysis is assigned an STL Edison job number. The job number is the primary key that is used to retrieve information pertinent to the sample submission.

2.1.2. A job folder is created when a group of samples is received. It is kept in the Document Management Office while the analysis is in progress. Internal and external sample control documents are kept in this folder including Chain of Custody documents. Instrument data specific to the samples in the group is also deposited in the job folder. These documents specify the dates and times the samples were prepared or analyzed which provides a cross-reference to the appropriate laboratory logbooks.

2.1.3. Individuals wishing to retrieve job specific information need only to look at the job folder. If further information is required, the laboratory notebooks may be located by the cross-reference provided in the job folder. The instrument run logs also give an indexed key to archived analytical data stored on cassette tape or diskette. Other documents include the following:

Laboratory Notebooks
Extraction Logs
Preventive Maintenance Logs
Balance Logs
Standards Prep Logs

2.1.4. After a job is completed and the final data report is sent to the client, the entire job folder is sequentially archived for not less than five years and is easily accessible if information is required at a later date.

2.2. Laboratory Logbooks

2.2.1. Separate logbooks are dedicated to separate procedures, functions and instruments. The information contained in the logbooks is unique to the operation to which it is dedicated.

2.2.1.1 *Laboratory Notebooks* provide the most basic and fundamental information about sample preparation and analysis. They contain at a minimum, the date of analysis, sample number, job number and signature of the responsible party.

2.2.1.2 *Extraction Logs* provide all the specific information about the extracted samples. This information include sample ID, Job number, matrix, Analytical method, Prep method, QA Batch, Blank associated, QA performed (if any), Analyst's initials, Extraction date, Extraction solvent and Lot number, Extraction volume, sample initial and final volume/weight, spike amount and the witness's initials, soxtherm position, pH of the sample, surrogates Lot number and amount spiked, and comments such as problems on the procedure or the sample itself.

2.2.1.3 *Preventive Maintenance Logs* provide various instrument maintenance information including the date, action taken, serial number for the replacement parts (noted on the comments section) and the analyst's initials.

2.2.1.4 *Balance Logs* contains calibration records, which include the date of calibration, observed weight values (up to three class S weights), special comments such as balances being taken out for service, and the initials of the person performing the calibration.

2.2.1.5 *Standard Prep Logs* contain fundamental information about the preparation of standards. This includes the Standards being prepared, the date of preparation, date of expiration, manufacturer's, initial concentration (for a secondary dilutions), final volume, solvent used and its Lot number, STL Lot number, initials of the person preparing the standards, and a second reviewer's initials.

2.2.2. The logbooks are maintained in a sequential manner. They are bound, paginated, filled out in black pen only and subject to signature authentication procedures. Logbooks that become filled are archived in the laboratory.

3. CORRECTIVE ACTIONS AND CONTINGENCIES FOR HANDLILNG OUT-OF-CONTROL OR UNACCEPTABLE DATA

Data that fails to meet minimum acceptance criteria will be annotated(flagged)with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Report will be initiated in order to provide for investigation and follow-up. For further corrective actions and contingencies for handling out-of-control or unacceptable data see *"Out of Control Events Corrective Actions"* SOP.

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