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ENVIRONMENTAL RESOURCES MANAGEMENT – NORTH CENTRAL, INC.

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REMEDIAL ACTION
SAMPLING AND ANALYSIS PLAN
PART II
QUALITY ASSURANCE PROJECT PLAN
VOLUME 2 - APPENDICES
REVISION: 1

ENVIRO-CHEM SITE
ZIONSVILLE, INDIANA

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PART II - QUALITY ASSURANCE PROJECT PLAN
VOLUME 2 - APPENDICES

ENVIRO-CHEM SITE
ZIONSVILLE, INDIANA

DECEMBER 10, 1991

REVISION: 1

PREPARED FOR:

ENVIRO-CHEM TRUSTEES

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APPENDIX A

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

APPENDIX A

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORMATION

TABLE OF CONTENTS

A.1	Warzyn Engineering, Inc. - Standard Operating Procedure for Tin Analysis
A.2	Standard Operating Procedure for 1,1-Dichloroethane Analysis
A.3	Chain-of-Custody Procedures
A.4	Standard Operating Procedures for Production Planning and Control
A.5	Data Processing Procedures
A.6	Corrective Action and Performance Audits Procedures
A.7	Facilities, Equipment, and Services

APPENDIX A.1

WARZYN ENGINEERING, INC.

STANDARD OPERATING PROCEDURE FOR TIN ANALYSIS

APPENDIX A.1

WARZYN ENGINEERING, INC.

STANDARD OPERATING PROCEDURES AND OTHER INFORAMTION

TABLE OF CONTENTS

Part I: Acid Digestion for Aqueous Samples and Extracts

Part II: Inductively Coupled Plasma - Atomic Emission Spectrometric Method

Part III: Additional Information

Effective: 7-2-91

ACID DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS
ICP/Flame-AA

Scope and Application: This acid digestion is applicable to all aqueous sample matrices. A nitric/hydrochloric acid digestion is used to prepare all samples which are to be analyzed by flame atomic absorption spectroscopy (flame-AA) or by inductively coupled plasma spectroscopy (ICP). A nitric acid/hydrogen peroxide digestion is used to prepare samples for analysis by graphite furnace atomic absorption spectroscopy (GFAA).

Method: Nitric acid/hydrogen peroxide and nitric/hydrochloric acid digestions

Reference: "Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Aqueous samples must be acidified with concentrated nitric acid to pH <2. Set up digestion as soon as possible; digested sample must be analyzed within 6 months.

Reagents and Apparatus:

1. Hot plate
2. 250 mL beakers
3. 100 mL graduated cylinders
4. Class A volumetric glassware
5. Deionized (D.I.) water
6. Instra-analyzed nitric acid, or equivalent
7. Distilled nitric acid (GFAA digestion only)
8. Instra-analyzed HCl acid, or equivalent
9. Stock and standard metal solutions
10. Whatman #42 filter paper
11. Glass or plastic funnels
12. Watch glasses
13. 30% Hydrogen peroxide

Reagent Preparation:

1. **Intermediate and working metal solutions:** Refer to the specific metal SOP for instructions on preparation.

2. **1:1 Hydrochloric acid (HCl):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HCl and mix.

3. **1:1 Nitric acid (HNO_3):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HNO_3 and mix.

Notes:

1. A separate digestion is required for mercury analyzed by the AA-Cold Vapor technique. (See "Mercury Digestion-Aqueous Samples")

2. All samples, duplicates, and spikes, as well as any required prep or digested blanks and standards, must be carried through the digestion procedure.

3. If samples boil or go to dryness (**any** dry spots on the bottom of the beaker) at any time during the digestion, some of the analyte may have been lost. The digestion must be discarded and the affected samples must be reprepared.

4. If elevated analyte levels are expected, the spike concentration may be increased accordingly.

Procedure:

Digestion Procedure for Flame-AA and ICP:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 2.0 mL of 1:1 HNO_3 and 10 mL of 1:1 HCl.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for analysis using the AA-flame or ICP methods.

Digestion Procedure for GFAA:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 1.0 mL of 1:1 HNO_3 and 2.0 mL of 30% H_2O_2.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for GFAA analysis.

Quality Control:

1. A digested blank and standard (spiked blank) must be included with each batch of samples that is digested. The blank is a check for possible contamination during the digestion process; the standard is a check for possible analyte loss during digestion.

2. A matrix spike and duplicate must be prepared, at a minimum, for every 10 samples digested. If fewer than 10 samples are digested a spike and duplicate are still required.

INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROMETRIC METHOD*

Scope and Application: Metals in solution can be readily analyzed by atomic emission using an inductively coupled plasma. Dissolved metals are determined in filtered and acidified samples. Total metals are determined in acidified, but unfiltered samples. Appropriate steps must be taken in all analyses to ensure that potential spectral interferences are taken into account.

Method: Inductively coupled plasma - atomic emission.

Reference: "Methods for Chemical Analysis of Water and Wastes", Method 200.7 EPA 1984.

"Inductively Coupled Plasma - Atomic Emission Spectroscopy", Method 6010, SW-846, November 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

"Instructions: Plasma 40 Emission Spectrometer", Perkin-Elmer, 1987.

Sample Handling: Acidify aqueous samples with concentrated nitric acid to pH <2. All samples must be digested prior to analysis (refer to appropriate digestion procedure). All samples must be analyzed within 6 months of sampling date.

Reagents and Appartus:

1. Plasma 40 Perkin-Elmer ICP Spectrometer
2. Argon (liquid: "high purity" or gaseous: "prepurified" grade)
3. Stock and intermediate metal standard solutions
4. EPA, ERA, or other reference standard solutions
5. Nitric acid, conc. (instra-analyzed or equivalent grade)
6. Class A volumetric glassware
7. Deionized water
8. Disposable 15 mL centrifuge tubes
9. 100 uL Eppendorf pipetter
10. 5 or 10 mL Oxford pipetter
11. Yttrium or Scandium stock solution
12. IBM AT Computer or equivalent
13. Epson 800 printer

Procedure:

Instrument Set-Up Procedure for Plasma 40:

1. Turn ON power switch if necessary (routinely left ON throughout week). Allow 1 hour for RF generator to warm up and electronic and optical components to achieve thermal equilibrium.

*Note: Only the results of the analysis for tin will be reported for this project.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:22AM P.02

2. Perform daily maintenance as specified in Maintenance Procedures: check pump, pump tubing, and nebulizer tips for wear, cleanliness, etc.

3. Turn on argon at tank. The first three indicator lights on the ICP (Power, RF ready, Interlock) should light.

4. Lock pump tubing in place, raise torch to the "ignite" position, and press "RF on".

5. When plasma ignites, lower torch to the run position (the injector tip should be even with or just below the bottom of the lowest RF coil).

6. Turn on pump and aspirate rinse water*. Allow plasma to stabilize 30 to 40 minutes before starting analysis.

Computer Start-Up Procedure:

1. Turn computer and printer power on (the computer will automatically start with a memory check).

2. Type CD ICP and press Return to enter the ICP directory. Then type ICP and press Return again to load software (approximately 10-15 seconds).

3. Perform a BEC check as specified in Maintenance Procedures. The BEC and CV values must be within the specified range before any analysis is done.

Sample Analysis:

1. Before starting analysis, for each element to be analyzed:

   a. Press F1 to select the Element mode, type the appropriate element file name and press Alt F9 to retrieve it from Library.

   b. Press F8 to select Spectrum mode.

   c. Analyze a single element standard at approximately 2-10X the IDL.

   d. Analyze the ICS AB solution.

   e. Analyze 1-3 samples representative of the digestion set.

   f. Compare the displayed spectra to check for spectral interferences. Reset background correction points as needed. If there are overlapping peaks or other spectral interferences present, an alternate wavelength or interelement correction must be used.

   g. Press F8 to leave the Spectrum mode. If wavelength calibration or background correction points were changed, press F9 to save the changes.

* Rinse water should be D.I. water with a small amount of liquid detergent (such as Liquinox or Whisk) added to improve wetting of tubing and spray chamber. Approximately 1-2 mL of soap per 500 mL water should be sufficient.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:22AM P.03

2. To store a list of sample labels to be used for the analytical run, select Report mode (F3), then ID/Wt mode (F8). Enter a file name, type in N (no) in the field for raw emission counts, mg/L for uncorrected units, and leave the corrected units field blank. Enter sample labels in the sample ID field in the exact order of analysis; include all check standards, QC samples, etc. If it is possible that additional samples may be added to the end of an analytical run (dilutions, post-digestion spikes, linear range standards, etc.) add additional sample labels to the ID/Wt file in the form of single letters (A, B, C, etc.) and manually write in the correct sample labels after the analytical run is completed. Alternatively, a new ID/Wt file may be created after the analytical run is completed (in this case the raw data must be reprinted with the new file by selecting Report Format 1 (F5) in the report mode and responding to the prompts). Save ID/Wt files by pressing F9 (to library).

3. Press F2 to select the Method mode.

4. Type the method file name and press Alt F9 to retrieve the desired method panel from Library, or create a new panel using existing element files. Standard conditions are 35 second read delay, 2 replicates per sample, report format #2 and a data file name composed of the date (mmdd) and a sequential letter identifier (e.g. 0123B for the second analytical run on Jan 23). An internal standard (usually yttrium) must be included in any method. Background correction points are already included in each element file.

5. Add yttrium (or scandium) stock solution (1000 mg/L) as an internal standard to all standards, blanks, and samples in a ratio of 0.1 mL yttrium stock to 10 mL sample. This allows automatic correction for matrix differences in viscosity, surface tension, etc.. If the autosampler is to be used, samples can be pipetted directly into 15 mL centrifuge tubes. Otherwise mix sample and yttrium in small disposable beakers.

If autosampler is used: *

1. If the autosampler is to be used, load sampler starting with the calibration standards in order of decreasing concentration (highest concentration first, calibration blank last).

2. Start automatic run (F5). Respond to the prompts that appear at the bottom of the screen:

   a. "Press start function key to begin this analysis": press F5

   b. "Enter ID/Wt file": type ID/Wt file name and press Return.

   c. "Do you wish to rinse between tubes (Y or N)": type Y and press Return. N may be selected only for clean samples where no carry-over problems are anticipated. Always rinse between samples when analysis is following CLP protocols, or analyzing for Sb, Cr, or Zn.

* Note: The ICP autosampler will be used for all analytical runs except in the event of mechanical failure.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:23AM P.04

d. "Enter position of the last sample in tray": type appropriate number and press Return (you may wish to enter a number several positions past the last sample to allow room for the addition of necessary dilutions, etc. at the end of the run).

e. "Do you wish to re-standardize (Y or N)": type N or Y and press Return. N is usually selected. Y will allow restandardization of the instrument during an automatic run but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional calibration standards.

f. "Do you wish to wavelength calibrate during the analysis? (Y or N)": type N or Y and press Return. N is usually selected. Y will allow recalibration of all wavelengths used in the current method before analysis is started but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional wavelength calibration standards. The system will then begin the analysis.

3. When the analysis is complete press F2 to select Method mode before exiting software to ensure the data file is stored permanently. Then set up the next panel, return to Report mode to set up a new ID/Wt file or reprint data, or press ESC to exit the ICP software.

If samples are to be run manually:

1. Press F2 to start a manual run and respond to prompts to calibrate instrument: Press F6 (Standard), aspirate the first calibration standard and press Return. At the prompt, aspirate the next standard(s) and press Return. When all calibration standards have been analyzed press F5 (Blank) aspirate the calibration blank and press Return. This completes the instrument calibration.

2. To analyze samples, type in sample label if needed, press F7 (Sample), aspirate sample and press Return. Repeat with all samples in the run.

Computer Shut-Down Procedure:

1. When analysis is complete press F2 (Method mode). At the message "Do you wish to quit method"? Type Y.

2. Press "ESC". At the message "Do you wish to quit method?" Type Y.

3. Turn off computer power switch.

4. Turn off printer.

Caution: Never turn off computer power while still using ICP software. This can cause partial loss of files and other errors.

Instrument Shut-Down Procedure:

1. Aspirate a dilute nitric acid solution (approx. 10%) for 1 to 2 minutes to clean sample introduction system.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:27AM P.12

2. Aspirate D.I. water for 5 minutes to rinse system thoroughly.

3. Turn off pump and release pump tubing.

4. Press "RF off" to extinguish plasma.

5. Shut off argon flow at tank.

6. If the ICP will not be used for 2 days or more, turn off ICP power switch. Otherwise, leave the ICP power ON.

Quality Control:

1. Establish a standard curve with the appropriate calibration standards plus a blank. Record the emission count for the internal standard in the ICP log book. The emission count should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check pump tubing, nebulizer tips, nebulizer flow, wavelength calibration, etc.).

2. **The first analyses for each analytical run are, in order:**

   a. Initial calibration verification standard (ICV)

   b. Initial calibration blank (ICB)

   c. Initial standard at 2X the CRDL (CRI). Note: The CRI is not necessary for Ca, Mg, Na or K.

   d. Initial interference check sample, solution A (ICSA).

   e. Initial interference check sample, solution AB (ICSAB)

   f. Laboratory control standard - an ERA, EPA, or other reference standard digested with the sample set (LCS)

To continue with sample analyses, the ICV must be within 90-110% of the true value, the ICB must be less than the CRDL, and the LCS and ICS solutions must be within 80-120% of the true value. If these QC criteria are not met, discontinue the analytical run and perform necessary troubleshooting.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are to be analyzed, a duplicate and spike are still required. Duplicates and spikes are to be within required control limits or the data must be flagged appropriately (N for spikes, * for duplicates). Additionally, if a digested spike is outside required control limits, a post-digestion spike must be analyzed for that sample.

4. For each sample batch (same matrix and project) one sample must be analyzed at an additional 5X dilution for the ICP serial dilution analysis (L). If the original sample concentration is at least 50X above the IDL, the serial dilution must agree within 10% of the original sample concentration or data for all associated samples must be flagged appropriately (E).

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:23AM P.05

5. A continuing calibration verification standard (CCV) and blank (CCB) are to be analyzed, at a minimum, after every 10 analyses. If less than 10 analyses are performed, a CCV and CCB are still required. The last samples analyzed in the run are to be a CCV and CCB. The CCVs must be within 90 - 110% of the true value or the samples run after the last acceptable calibration standard are to be reanalyzed.

6. At the end of each analytical run, but before the final CCV and CCB, the CRI, and ICS solutions A and AB are to be reanalyzed. The ICS must be within 80-120% of the true value or the samples run after the last acceptable calibration verification standard are to be reanalyzed.

7. Refer to the appropriate Quality Assurance Project Plan (QAPP) for project specific QC information (additional QC requirements, matrix spike and duplicate control limits, etc.).

8. Detection limit verifications and linear range analyses must be performed each quarter. Interelement correction factors are to be determined annually at a minimum. Interelement correction factors must be recalculated on an analyte and wavelength specific basis any time background correction points are changed in an element file. Additionally, for greatest accuracy, interelement correction factors should be re-determined for any analytical batch that is expected to have high concentrations of common interferents (e.g. any soil, sediment, sludge, or leachate matrix).

Daily Maintenance Procedures - Plasma 40

1. **Pump rollers:** With the pump on, feel along the bottom of the pump to determine that all the rollers are turning smoothly with no resistance or pulling. If a "sticky" roller is found a service call must be placed to Perkin-Elmer to correct the problem. A sticky roller will cause rapid deterioration of pump tubing resulting in erratic results.

2. **Pump tubing:** Check pump tubing for excessive stretching, soft or flattened spots. This can cause irregular or diminished sample flow resulting in reduced sensitivity and lack of precision in sample results. When pump tubing is changed, (usually after 6-8 hours of use) it is necessary to trim ends of the new tubing so the length from the black stops to the end of the tubing is kept constant. Failure to trim tubing ends can cause imprecise results due to a longer sample read delay.

3. **Nebulizer tips:** Remove nebulizer end cap and check nebulizer tips visually and with the cleaning wire for clogs, salt build-up or other deposits. Follow the instructions in the Plasma 40 operating instructions for replacing nebulizer tips if necessary (Part 2, pg 3-8). Used tips may be cleaned by soaking overnight in 10% nitric acid followed by thorough rinsing with D.I. water. Finally, with the argon on, aspirate water and observe the spray pattern. The nebulizer should produce a fine, even mist with no large droplets with the direction of the spray approximately perpendicular to the face of the end cap (should not deviate more than about 20°). If the spray pattern looks uneven, "bent", or is pulsing excessively, recheck pump tubing and review nebulizer maintenance to correct the problem.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:24AM P.06

4. **BEC check:** This is an indication of how well the ICP system is performing. After the plasma has been ignited and allowed to stabilize for 30-40 minutes perform the following steps:

   a. At the DOS prompt type ICP and press Return to load software.

   b. Type MnBEC and press Alt F9 to retrieve this method from Library. Press F6 to start a manual run.

   c. Press F6 again (standards), aspirate a 1.0 mg/L Mn standard and press Return. The ICP will analyze 10 replicates of this standard. The Coefficient of Variance (CV) for these readings should be <2.0. If a higher values is obtained a sample introduction or instrument calibration problem is indicated. Check pump tubing and wavelength calibration for Mn and repeat the analysis. Record the CV in the maintenance log book.

   d. Press F5 (blank), aspirate a blank, and press Return to complete the calibration.

   f. Turn off the torch (RF off), aspirate D.I. water, and press F7 (sample). The resulting concentration should be $\leq |0.040|$. If a higher value is obtained, a problem with the sample introduction system is indicated. Review maintenance and, if the problem cannot be corrected, place a service call with Perkin-Elmer. Record the BEC in the maintenance log book.

   i. Re-light the torch and press ESC to end the manual run. Allow the plasma to stabilize 10-15 minutes before beginning any analysis.

5. **Final rinse:** When analysis for the day is complete, aspirate dilute (approx. 10%) nitric acid for one or two minutes followed by D.I. water for approximately 5 minutes. This will help prevent deposits from building up in the sample introduction system. Remember to release pump tubing when completed.

Weekly Computer Backup:

1. Once a week data files should be copied to floppy disks and deleted from the hard disk. Data files on floppy disks should be saved for one year.

2. Periodically (every 1-3 months depending on work volume), files should be reviewed, old files deleted and the entire system backed-up.

Other Maintenance:

1. Occasionally, additional maintenance will be necessary to correct problems arising from time and wear on the system. Any additional maintenance performed (including P.E. service calls) should be listed in the maintenance logs. These include periodic cleaning of the torch assembly, inspection of O-rings in torch assembly, and wavelength recalibration. Generally, these procedures will only be performed in response to observed problems. Refer to the Plasma 40 operating manual for specific directions.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:24AM P.07

ICAP CALIBRATION STANDARDS

Element	Wave-length	Detection Limit(ug/L)	Cal. Std. 1 (ug/L)	Cal. Std. 2 (ug/L)	Cal. Std. 3 (ug/L)	ICV (ug/L)	CCV (ug/L)
Al	237.335	50	20,000	400	---	2500	4000
Al	396.152	50	20,000	400	---	2500	4000
Sb	206.833	50	2000	500	250	1000	1000
Ba	233.527	10	10,000	200	---	500	2000
Be	313.107	5	1000	20	---	250	200
Cd	228.802	5	1000	50	---	500	200
Cd	214.438	5	1000	50	---	500	200
Ca	317.933	1000	200,000	10,000	---	10,000	80,000
Cr	267.716	10	10,000	500	200	1000	2000
Cr	205.552	10	10,000	500	200	1000	2000
Co	238.892	50	10,000	200	---	2500	2000
Co	228.616	10	10,000	200	---	2500	2000
Cu	324.754	10	10,000	100	---	1000	2000
Cu	224.700	20	10,000	100	---	1000	2000
Fe	238.204	20	20,000	200	---	1000	4000
Pb	220.353	100	10,000	500	---	5000	2000
Pb	216.999	100	10,000	500	---	5000	2000
Mg	285.213	1000	100,000	5000	---	10,000	40,000
Mn	257.610	10	10,000	100	---	500	2000
Ni	352.454	20	10,000	100	---	1000	2000
Ni	232.003	20	10,000	100	---	1000	2000
Ag	338.289	10	1000	---	50	500	200
Na	330.237	2000	100,000	5000	---	20,000	40,000
Sn	189.989	200	10,000	1000	---	2500	5000
V	292.402	50	10,000	500	250	2500	2000
Zn	213.856	10	10,000	100	50	500	2000

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:25AM P.08

ICAP CALIBRATION STANDARDS

ICAP calibration standards are prepared from both multi-element stock solutions purchased from SPEX Industries (custom mixed standards) and single element stock solutions from VWR and Baxter (Ricca or Mallinckrodt as available).

XWE-1	XWE-2	XWE-3a	XWE-4a
2000 mg/L Fe	20,000 mg/L Ca	1000 mg/L Cr	2000 mg/L Al
1000 mg/L Cu	10,000 mg/L Mg	1000 mg/L Pb	1000 mg/L Ba
1000 mg/L Mn	10,000 mg/L Na	1000 mg/L V	1000 mg/L Co
1000 mg/L Ni		100 mg/L Cd	100 mg/L Be
1000 mg/L Zn			100 mg/L Ag

XWE-6a

500 mg/L Pb
250 mg/L Co, Al
100 mg/L Cu, Ni, Fe, Cr
50 mg/L Ba, Cd, Ag, Zn, Mn

Single Element Stock Solutions 1000 mg/L

Sb Ag
Be Na
Ca V
Cr Zn
Mg Sn

Calibration Standard #1:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, Zn:** Into a 1 L volumetric flask, add 500 mL of de-ionized (D.I.) water and 50 mL of concentrated HCl. Pipet 10 mL each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sb stock solution. Dilute to volume with D.I. water.

3. **For Sn:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 5.0 mL of 1000 mg/L Sn stock solution. Dilute to volume with D.I. water.

Calibration Standard #2:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Na, V, and Zn:** First prepare 10X dilutions each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Then, into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 1.0 mL of XWE-1 (10X dilution), 5.0 mL of XWE-2 (10X dilution), 5.0 mL of XWE-3a (10X dilution), and 2.0 mL of XWE-4a (10X dilution). Dilute to volume with D.I. water.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:26AM P.09

2. **For Sb:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 50 mL of Sb Calibration Standard #1 and dilute to volume with D.I. water.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 20 mL of Sn Calibration Standard #1 and dilute to volume with D.I. water.

Calibration Standard #3:

1. **For Cr, Ag, V and Zn:** First prepare intermediates as follows:

   50 mg/L Ag and Zn: Into a 100 mL volumetric flask, add 10 mL of 1:1 HCl. Pipet 5.0 mL each of single element Ag and Zn stock solutions and dilute to volume with D.I. water.

   100 mg/L Cr: Into a 100 mL volumetric flask pipet 10.0 mL of single element Cr stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   50.0 mg/L V: Into a 100 mL volumetric flask pipet 5.0 mL of single element V stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   Then, into a 500 mL volumetric flask, add 250 mL D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 100 mg/L Cr intermediate, 0.5 mL of 10 mg/L Ag-Zn mixed intermediate, and 2.5 of 50 mg/L V intermediate. Dilute to volume with D.I. water.

2. **For Sb:** Into a 100 mL volumetric flask, add 50 mL of D.I. water and 10 mL of 1:1 HCl. Pipet 50 mL of Calibration Standard #2 and dilute to volume with D.I. water.

Initial Calibration Verification:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 10 mL of XWE-6a, 10 mL of 1000 mg/L Mg stock, 10 mL of 1000 mg/L Ca stock, and 20 mL of 1000 mg/L Na stock solutions. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 0.5 mL of 1000 mg/L Sb stock and dilute to volume with D.I. water.

3. **For Sn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 2.5 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:26AM P.10

Continuing Calibration Verification Standard:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL D.I. water and 50 mL of concentrated HCl. Pipet 2.0 mL each of XWE-1, XWE-3a, XWE-4a, and 4.0 mL of XWE-2. Dilute to volume with D.I. water.

2. **For Sb:** Use Sb Initial Calibration Verification Standard.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL D.I. water and 10 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

FROM:KONICA FAX TO: 919 248 6786 AUG 2, 1991 10:27AM P.11

Part III: Additional Information

1. Working Linear Range: The working linear range is specific to each element and wavelength. Linear ranges are determined quarterly, at a minimum, and any time method or calibration parameters are changed. Linear range information is reported on Form 12 of the final data package.

2. Interferences and Corrective Actions: The possible presence of spectral interferences (including interelement interferences) is evaluated for each batch of samples ("batch" refers to samples of the same project, matrix, and digestion set) and corrected as outlined in the Sample Analysis section of Part II. The presence and extent of other matrix and/or chemical interferences is checked through the required matrix-specific quality control samples (spikes, duplicates, serial dilutions). Any affected data is handled as outlined in the 3/90 SOW, including flagging of data, redigestion of samples, and/or reanalysis as appropriate.

3. Safety Precautions: Safety precautions are discussed in detail in the laboratories' Health and Safety Manual and in their Basic Laboratory Requirements.

4. Storage of Reagents and Standards: All reagents and standard solutions are stored at room temperature. Stock solutions are kept for no longer than one year. While there are no set guidelines for the shelf life of mixed-element standards, these standards are prepared in quantities sufficient for approximately two months' use. No standards are kept for longer than one year. Alternate source initial calibration standards and laboratory control standards are required for each analytical run and insure that calibration standard solutions are accurate and stable.

5. Data Treatment: ICP data is reduced automatically by the ICP operating and reporting software which runs on a dedicated Northgate PC (IBM compatible). Data is simultaneously printed and stored to disk during the analytical run. Raw data includes analyst initials, lab sample number, analyte, date and time of analysis, analyte

concentration in mg/L (for both replicate instrument readings), and the average concentration, standard deviation, and coefficient of variance for each sample. Final reports are generated from raw ICP data files which are downloaded to a Telecation software package. This database and report package includes all algorithms necessary to convert the raw data to final reportables required under the SOW.

6. Data Deliverables: Data deliverables will include the following:
   - Laboratory case narrative,
   - Forms 1-14 as required under the 3/90 SOW,
   - All laboratory digestion/sample preparation logs,
   - Computer printouts of all raw data, and
   - Copies of all chain-of-custody records.

7. Quality Control Requirements: The QC requirements of the 3/90 SOW will be used for this project. These requirements are as follows:
   - 75-125% recovery for matrix spikes, except where the sample concentration is greater than 4 times the spiking level;
   - 20% relative percent difference for duplicate analyses, where sample concentration is greater than 5 times the CRDL; and
   - ± CRDL for duplicate analyses where sample concentration is less than 5 times the CRDL.

APPENDIX A.2

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR 1,1-DICHLOROETHANE ANALYSIS

INTRODUCTION TO METHOD 8010 SOP

CompuChem Laboratories' SOP for "Analysis of Purgeable Halogenated Volatile Organic Compounds in Water by Method 8010" follows this introduction. Several additional items regarding the use of this method for this project are listed below.

1. Parameter to be measured: 1,1-Dichloroethane in water is the only analyte of interest.

2. Interferences and corrective actions: Contamination by carryover can occur whenever high level and low level samples are sequentially analyzed. To prevent carryover, analyze one or more instrument blanks to check and clean the system. In extreme situations, the purge and trap system may require dismantling and cleaning.

3. Safety precautions: According to the Federal Register, the following compounds have been tentatively classified as known or suspected carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. A gas respirator should be worn when handling samples with high quantities of these compounds.

4. Frequency of calibration: every 24 hours.

5. Data treatment: The following equations are used to quantify the data.

$$\text{Response Factor (RF)} = \frac{\text{area of compound to be measured}}{\text{conc. of the compound to be measured in the standard}}$$

Two internal standards are used in Method 8010: Trichlorofluoromethane (TCFM) and bromofluorobenzene (BFB).

$$\text{TCFM Correction Factor (CF)} = \frac{\text{area of TCFM in sample}}{\text{(avg. RF of TCFM from multipoint/10)}}$$

$$\text{BFB Correction Factor (CF)} = \frac{\text{area of BFB in sample}}{\text{(avg. RF of BFB from multipoint/10)}}$$

Compound Quantitation equation =

(1) $$\frac{\text{area of compound in sample}}{\text{(avg. RF of compound in sample * TCFM CF)}}$$

(2) $$\frac{\text{area of compound in sample}}{\text{(avg. RF of compound in sample * BFB CF)}}$$

Equation (2) is used for the quantitation of 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. All other 8010 compounds are quantitated using equation (1).

$$\text{TCFM \% Recovery} = \frac{\text{TCFM CF}}{\text{BFB CF}} * 100$$

$$\text{BFB \% Recovery} = \frac{\text{BFB CF}}{\text{TCFM CF}} * 100$$

All quantitations are rounded to two significant figures.

6. Reporting: Only the results for 1,1-Dichloroethane will be reported. A list of deliverables is attached.

DELIVERABLES CODE FOR REPORT FORMAT

Style 9 includes:

- Cover letter
- Table of Contents
- Chronicle (including QC summary)
- Case Narrative
- Method Reference
- QA Notices (if applicable)
- Chain-of-Custody (if received)
- Compound List
- RIC (sample)
- Quant. Report
- Spectra
- Library Search Form IV (if applicable)
- Library Search Spectra (if applicable)
- Blank Compound List
- RIC (blank)
- Quant. Report
- Spectra
- Matrix Spike/Matrix Spike Duplicate
- RIC (spikes)
- Quant. Report
- Tuning Summary (for sample, blank & spikes)
- Calibrations (Initial & Continuing for sample blank & spikes)
- RIC (standards: includes, in order, samples, blank & spike)
- Quant. Report (sample, blank & spike)

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ERM-NORTH CENTRAL, INC.

Instrument Procedure 153, 154, 157, 165: Analysis of Purgeable Halogenated Volatile Organic Compounds in Water by Method 8010

1.0 Summary

Method 8010 provides gas chromatographic conditions for the detection of halogenated volatile organic compounds. Purgeable halogenated compounds are extracted from water by a room temperature purge and trap extraction. The trap is desorbed onto a 105-m, 0.53mmID capillary column, and analytes are detected by an Electrolytic Conductivity Detector (ELCD) which is mounted on a gas chromatograph. The gas chromatograph is temperature programmed, complete with a cryogenics (liquid nitrogen) system, to separate the target compounds. A surrogate is used to measure the sample recovery. Target compounds are quantitated with a multipoint and external standard calculation.

This procedure meets the requirements set forth in the following References for Approved Methods: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods; SW-846, 3rd edition, revised September 1986; and U.S. EPA Federal Register Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Final Rule, Interim Final Rule, and Proposed Rule. Vol.49, Part 209.

2.0 Target Compound List

Analyte	CAS Number
chloromethane	74-87-3
vinyl chloride	75-01-4
bromomethane	74-83-9
chloroethane	75-00-3
1,1-dichloroethene	75-35-4
methylene chloride	75-09-2
trans-1,2-dichloroethene	156-60-5
1,1-dichloroethane	75-34-3
chloroform	67-66-3
bromochloromethane	74-97-5
1,1,1-trichloroethane	71-55-6
carbon tetrachloride	56-23-5
1,2-dichloroethane	107-06-2
trichloroethene	79-01-6
1,2-dichloropropane	78-87-5
bromodichloromethane	75-27-4
dibromomethane	74-95-3
2-chloroethyl vinyl ether	110-75-8
cis-1,3-dichloropropene	10061-01-5
trans-1,3-dichloropropene	10061-02-6

Analyte	CAS Number
1,1,2-trichloroethane	79-00-5
tetrachloroethene	127-18-4
dibromochloromethane	124-48-1
1,2-dibromoethane	106-93-4
chlorobenzene	106-90-7
1,1,1,2-tetrachloroethane	630-20-6
bromoform	75-25-2
1,1,2,2-tetrachloroethane	79-34-5
1,2,3-trichloropropane	96-18-4
bromobenzene	108-86-1
2-chlorotoluene	95-49-8
4-chlorotoluene	106-43-4
1,3-dichlorobenzene	541-73-1
1,4-dichlorobenzene	106-46-7
1,2-dichlorobenzene	95-50-1

3.0 Interferences and Safety

3.1 Interferences

Methylene chloride (which is used as an extraction solvent in other laboratories within the facility) is occasionally detected at low levels in blanks and samples analyzed by Methods 601 and 8010. CompuChem has taken measures to minimize this contamination, including physically isolating the volatile instrumentation lab, polishing incoming air through activated carbon filters built into the air handling system, maintaining a positive air pressure within the lab, and restricting access to the lab by personnel who use volatile solvents.

CompuChem has adopted an internal policy based on guidelines established by the EPA's Contract Laboratory Program which permits up to five times the detection limit for common contaminants in method blanks. The maximum methylene chloride contamination allowed in an 8010 aqueous method blank is 5 µg/l.

Contamination can also occur by carryover whenever high-level and low-level samples are sequentially analyzed. To prevent carryover, analyze one or more instrument blank to check and clean the systems. In extreme situations, the purge and trap system may need to be dismantled and cleaned.

3.2 Safety

According to the Federal Register, the following compounds have been tentatively classified as known or suspected carcinogens:

- carbon tetrachloride
- chloroform
- 1,4-dichlorobenzene
- vinyl chloride

Wear a gas respirator when handling samples with quantities of these compounds.

4.0 Sample Preparation Screen

All samples of unknown composition should be screened prior to analysis. Screening methods involve a 50:1 dilution followed by purge and trap analysis.

5.0 Procedure

5.1 Introduction of Sample

Put five milliters (ml) of sample into a 5- or 10-ml gastight syringe with Luerlok tip (Hamilton 1005 TEFLL or equivalent). Add five microliters (µls) of surrogate solution 418 with a 10-µl gastight syringe (Hamilton 1701N or equivalent). If the screen of the sample indicates that a dilution is required, add the following sample volumes to pure laboratory water spiked with surrogate.

Dilution	Vol. of sample(µl)	Vol. of Blank Water(ml)
2:1	2500	2.5
5:1	1000	4.0
10:1	500	4.5
20:1	250	4.8
50:1	100	4.9
100:1	50	5.0
200:1	25	5.0
250:1	20	5.0
500:1	10	5.0
1000:1	5	5.0

For dilutions greater than 1000:1, a serial dilution should be made. For example, a 10,000:1 dilution can be made by making a 100:1 dilution of a 100:1 dilution. The first dilution should be 100:1 or greater to minimize the risk of contaminating the syringes.

5.2 Sample Purge

The sample is injected into a purging vessel conforming to the specifications listed in the standardized methods. The sample is purged with helium at a flow of 40 ml/min for 12 minutes.

5.3 Sample Trap

The purge gas is swept through a trap containing 1.0 cm of OV-101, 7.7 cm of Tenax, and 15 cm of silica gel. An equivalent trap may be used if the trap adsorbs all the target compounds. The trap should be below 25° C during the purge procedure. The transfer line between the purge vessel and the trap should be kept above 80° C at all times.

5.4 Sample Desorption

During the desorb procedure, the target compounds are backflushed off the trap and then enter the carrier gas stream. The gas flow and trap temperature are controlled so that all the compounds are removed from the trap in as short a time as possible. When the GC is ready to start a run, the trap is preheated to 175° C then desorbed at 180° C. The backflush gas is helium flowing at 15 ml/min. The trap is desorbed for 4 minutes. The trap is then baked at 225° C for 18 minutes before being cooled to room temperature to remove water adsorbed on the silica gel during the heated purge.

Other desorb temperatures may be required with different trap material.

5.5 Chromatography

The following apparatus and conditions are used to chromatograph the target compounds.

5.5.1	GC:	Varian 3400 or equivalent
5.5.2	Injection Port:	Varian 1/4-inch packed column port at 200° C with Restek Uniliner and a Restek 0.80 OD insert set in the direct injection mode.

5.5.3 Column: Rtx-502.2 105 meter, 0.53-mm ID, 3.00-µm film (Restek #10910) or equivalent with the following temperature program: (All temperatures are in degrees C.) 0° for 4 min. Ramp to 90° at 3°/min., hold 2 min. Ramp to 130° at 2/min., hold 0 min. Ramp to 220° at 12°/min, hold 2 min. Total run time = 65.50 minutes.

The temperature program above is used to separate all 8010 compounds.

5.6 Detector Parameters

The O.I. Co. model 4420 Electrolytic Conductivity Detector is used for sample detection using the following parameters:

Mode:	Halogen
Make-up Gas:	Helium
Reaction gas:	Hydrogen
Reactor Temp:	850° C
Filter:	M
Range:	1 Volt
Attenuation:	1
Detector Solvent:	1-propanol

5.7 Initial Calibration and Quantitation

Response factor (RF) = area/std concentration
Sample Concentration = (area/mean RF) x dilution

Samples are quantitated using a five-level multipoint calibration with external standard quantitation. The following table lists the standard levels used to generate the multipoint response factors.

Compound	Concentrations (ug/L)				
	VL	L	M1	M2	H
chloromethane	4.0	8.0	16.0	24.0	32.0
vinyl chloride	4.0	8.0	16.0	24.0	32.0
bromomethane	4.0	8.0	16.0	24.0	32.0
chloroethane	4.0	8.0	16.0	24.0	32.0
1,1-dichloroethene	2.0	4.0	8.0	12.0	16.0
methylene chloride	2.0	4.0	8.0	12.0	16.0
t-1,2-dichloroethene	2.0	4.0	8.0	12.0	16.0
1,1-dichloroethane	2.0	4.0	8.0	12.0	16.0

Compound	Concentrations (ug/L)				
	VL	L	M1	M2	H
chloroform	2.0	4.0	8.0	12.0	16.0
bromochloromethane	2.0	4.0	8.0	12.0	16.0
1,1,1-trichloroethane	2.0	4.0	8.0	12.0	16.0
carbon tetrachloride	2.0	4.0	8.0	12.0	16.0
1,2-dichloroethane	2.0	4.0	8.0	12.0	16.0
trichloroethene	2.0	4.0	8.0	12.0	16.0
1,2-dichloropropane	2.0	4.0	8.0	12.0	16.0
bromodichloromethane	2.0	4.0	8.0	12.0	16.0
dibromomethane	2.0	4.0	8.0	12.0	16.0
2-chloroethyl vinyl ether	2.0	4.0	8.0	12.0	16.0
c-1,3-dichloropropene	2.0	4.0	8.0	12.0	16.0
t-1,3-dichloropropene	2.0	4.0	8.0	12.0	16.0
1,1,2-trichloroethane	2.0	4.0	8.0	12.0	16.0
tetrachloroethene	2.0	4.0	8.0	12.0	16.0
dibromochloromethane	2.0	4.0	8.0	12.0	16.0
1,2-dibromoethane	2.0	4.0	8.0	12.0	16.0
chlorobenzene	2.0	4.0	8.0	12.0	16.0
1,1,1,2-tetrachloroethane	2.0	4.0	8.0	12.0	16.0
bromoform	2.0	4.0	8.0	12.0	16.0
1,1,2,2-tetrachloroethane	2.0	4.0	8.0	12.0	16.0
1,2,3-trichloropropane	2.0	4.0	8.0	12.0	16.0
bromobenzene	2.0	4.0	8.0	12.0	16.0
2-chlorotoluene	2.0	4.0	8.0	12.0	16.0
4-chlorotoluene	2.0	4.0	8.0	12.0	16.0
1,3-dichlorobenzene	2.0	4.0	8.0	12.0	16.0
1,4-dichlorobenzene	2.0	4.0	8.0	12.0	16.0
1,2-dichlorobenzene	2.0	4.0	8.0	12.0	16.0

If the %RSDs over the five levels are less than 20%, then the mean response factor can be used to calculate the compound concentrations (see method 8000, section 7.4.2.2). Otherwise a calibration curve must be used to calculate the sample concentrations. It is expected that all compounds in method 8010 will have an RSD of <20%. If the RSD is >20%, the following items should be checked.

- If response factors have random variation, check for leaks in the purge vessels.
- If response factors increase with concentration, check for active sites in the chromatography system.
    - If response factors from direct injections increase with concentration the active sites are probably in the GC column or injector port.

- If response factors from direct injections remain consistent the active sites are probably in the purge and trap apparatus.

- If response factors decrease with concentration, check for contamination of the system by analyzing a blank.

5.8 Sample Identification

If single column analysis has been requested, a peak is identified as a target compound if its retention time is within the retention time window of a target compound. If a peak falls within multiple retention time windows, it is identified as the target compound with the closest relative retention time as compared to the surrogate.

If dual column analysis is requested, the sample is run on a dissimilar column if any peak is in a target compound retention time window during the first analysis.

The GC lab follows a policy that the identity of the peak is confirmed if the peak is in the retention time window for the same compound on both columns and the concentration of the compound on the second column is within - 50 to +100% of the concentration on the first column.

Exceptions to this rule can be made if there is reason to believe that matrix interference is affecting the concentration calculation on one or both columns. If an exception is made, the justification is documented, signed, and dated by the chemist performing the analysis in the form of a Laboratory Notice.

5.9 Setting Retention Time Windows

Setting accurate retention time windows is one of the most important procedures for the identification of target analytes. The procedure is covered by section 7.5 in method 8000 and paragraph 10.10 in method 601. Both methods use three times the standard deviation of three injections of a check standard over a 72 hour period. Method 601 says this procedure "can be used to calculate a suggested window size". Method 8000 says this procedure "will be used to define the retention time window". Both methods say the experience of the analyst should weigh heavily in the interpretation of chromatograms.

The procedure specified in the methods, determined by analyzing replicate check standards, will set a window that is too restrictive, increasing the risk of false negatives.

This is because analysis of check standards does not incorporate all the variables that can affect retention time.

Variables affecting retention times can be divided into two classes: system dependent and matrix dependent.

5.9.1 System-dependent variables

System dependent variables can be monitored and to a certain extent controlled by the GC chemist. Recent advances in GC technology have enhanced the ability of the chemist to control many system variables. The following system attributes have been found to affect retention times.

- Carrier gas flow rate: A consistent flow rate is important. The flow rate can be affected by changes in line pressure, leaks in the system, or changes in backpressure.

- Carrier gas density: Carrier gas density can have a surprising effect on retention time. In general, increasing the flow, molecular weight, and isothermal hold times will increase the effects of changes in density. The critical point where density is variable is at the flow controllers which are usually at "room" temperature. Therefore, precise control over the temperature of the lab can be important to retention time stability.

- Oven temperature: Oven temperature is the most important parameter affecting retention times. As such, it is an area where GC manufacturers have improved by producing very stable and uniform oven temperatures and heating ramps. In the volatile analysis, the Varian 3400 can achieve a stable isothermal temperature down to 35° C. Below that point, liquid nitrogen is required as a coolant. It is also critical that the oven temperature stabilizes for 2 minutes before the sample is desorbed. Therefore, the purge and trap should advance to "desorb preheat" after receiving a "GC ready" signal from the GC instead of relying on a thermocouple connection and minimum temperature cut-off.

- Desorb time: Controllable changes in the desorb time are caused by changes in the trap temperature and desorb gas flow. The desorb gas is controlled by the GC and is affected by all the parameters that affect carrier gas. The trap temperature is held at ambient levels during purge and ballistically heated during desorb preheat and desorb. Therefore, it is important that the ambient temperature remains stable. The heating profile of the trap is not important during desorb preheat because the target compounds are not moving during this period; however, it is important during desorb. Therefore, the preheat temperature must be close to the final desorb temperature.

5.9.2 Matrix-dependent variables

The sample matrix affects the desorb time and the column characteristics. Polar compounds in a matrix can coat the surface of the capillary liquid phase so that the target compounds skip along the column rather than plow through the phase.

Because of all these factors, each GC system has unique retention time windows. The window should be set at ± 1.5% of the retention time of each compound from each continuing calibration check standard.

6.0 Quality Control

6.1 Surrogates

The surrogates for method 8010 differ from method 601 because Trichlorofluoromethane(TCFM), a current surrogate for 601, is on the compound list for 8010. Because CompuChem does not validate this compound at this time (we do not see TCFM in water), TCFM and Bromofluorobenzene are used as the surrogates. TCFM is spiked at 10.0 µg/L, and historically generated 601 data indicate that the acceptance range is 76%-135%. Bromofluorobenzene is spiked at 10.0 µg/L and the acceptance range is 69%-123%. This range will be periodically updated as historical method performance data are accumulated.

6.2 Continuing Calibration Check Standards

Method 8010 appears to have very different requirements for check standard frequency and acceptance criteria from method 601. However, extensive conversations with the EPA have revealed that the SW-846 methods should have the same acceptance criteria as the 600-series methods. Method 601 (paragraph 7.5) requires a check standard every 10 samples, whereas method 8010 requires a check standard every working day. The check standard acceptance criteria is taken from Table 2 of method 601(page 43266), and Table 3 of method 8010(the two tables are identical), and not the +/-15% RSD in method 8000(pending EPA approval). This acceptance criteria only applies to the 601 compounds.

We will apply our historically generated laboratory acceptance ranges to the additional 8010 compounds. Our historically generated laboratory acceptance ranges are compared to the acceptance criteria listed in method 8010 (Table 3) in the following table:

Calibration Acceptance Criteria For Check Standards

Compound	8010 % recovery range	Lab % recovery range
chloromethane	D-193	1-193
vinyl chloride	28-163	31-170
bromomethane	D-144	1-180
chloroethane	38-150	39-139
1,1-dichloroethene	28-167	25-170
methylene chloride	25-162	25-165
t-1,2-dichloroethene	38-155	40-154
1,1-dichloroethane	47-132	50-130
chloroform	49-133	50-130
bromochloromethane	47-132[a]	47-132
1,1,1-trichloroethane	41-138	45-135
carbon tetrachloride	43-143	40-141
1,2-dichloroethane	51-147	50-150
trichloroethene	35-146	35-145
1,2-dichloropropane	44-156	46-160
bromodichloromethane	42-172	42-170
dibromomethane	42-172[a]	42-172
2-chloroethyl vinyl ether	14-186	15-200
c-1,3-dichloropropene	22-178	20-180
t-1,3-dichloropropene	22-178	20-185
1,1,2-trichloroethane	39-136	40-135
tetrachloroethene	26-162	20-170
dibromochloromethane	24-191	20-190
1,2-dibromoethane	24-191[a]	24-191
chlorobenzene	38-150	35-150

1,1,1,2-tetrachloroethane	8-184[a]	8-184
Compound	**8010 % recovery range**	**Lab % recovery range**
bromoform	13-159	10-170
1,1,2,2-tetrachloroethane	8-184	5-190
1,2,3-trichloropropane	60-140[a]	60-140
bromobenzene	60-140[a]	60-140
2-chlorotoluene	60-140[a]	60-140
4-chlorotoluene	60-140[a]	60-140
1,3-dichlorobenzene	7-187	5-190
1,4-dichlorobenzene	42-143	45-140
1,2-dichlorobenzene	D-208[b]	1-200

[a] Value taken from CompuChem's historically generated acceptance range.

[b] D = Detected

There is general agreement between the lab acceptance criteria ranges and the ranges from method 8010. The check standards will therefore be run after every 10 samples, and the acceptance criteria ranges will be from Table 3 in method 8010 with the exception of the non-method 601 compounds which will use our laboratory's acceptance range. When a check standard fails to meet the recovery criteria, an initial calibration must be performed. Failure of the check standard means analysis cannot continue until system maintenance is performed and the check standard passes criteria.

6.3 Blanks

Method 8000 specifies that one method blank be prepared per analytical batch (up to 20 samples). For method 8010, a blank will be run either once per day per instrument or once per analytical batch, whichever is more frequent. A method blank consists of laboratory pure water spiked with bromofluorobenzene (BFB) and trichlorofluoromethane (TCFM) at 5 µl per 5.0 ml of water. The method blank must not contain any target compounds above the reporting limit. Exceptions to this are common laboratory solvents (i.e., methylene chloride) which may be present in blanks at 5X the stated detection limit.

6.4 Matrix Spikes

As specified in method 8000 (paragraph 8.5), duplicate matrix spikes will be analyzed per analytical batch (up to 20 samples). If a spike fails the recovery criteria listed in the check standard acceptance ranges table above, then a

blank spike should be performed to verify that the GC system is in control. If the blank spike does not meet acceptance criteria, analysis cannot be continued until after system maintenance has been performed and a blank spike passes criteria. Then the duplicate matrix spikes should be re-analyzed. All samples associated with a failed blank spike must also be re-analyzed. Blank spikes must contain the same compounds at the same levels as the check standard.

7.0 Calculations

$$\text{Reponse factor (RF)} = \frac{\text{area of compound to be measured}}{\text{conc. of the compound to be measured in the standard}}$$

Method 8010 uses two internal standards, trichlorofluoromethane(TCFM) and bromofluorobenzene(BFB).

$$\text{TCFM correction factor(CF)} = \frac{\text{area of TCFM in sample}}{\text{(avg. RF of TCFM from multipoint/10)}}$$

$$\text{BFB correction factor(CF)} = \frac{\text{area of BFB in sample}}{\text{(avg. RF of BFB from multipoint/10)}}$$

Compound quantitation equation =

(1)

$$\frac{\text{area of compound in sample}}{\text{avg. RF of compound in sample x TCFM CF}}$$

(2)

$$\frac{\text{area of compound in sample}}{\text{avg. RF of compound in sample x BFB CF}}$$

Use equation (2) for the quantitation of 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. All other 8010 compounds are quantitated using equation (1).

$$\text{TCFM \% recovery calculation:} \quad \frac{\text{TCFM CF}}{\text{BFB CF}} \times 100$$

$$\text{BFB \% recovery calculation:} \quad \frac{\text{BFB CF}}{\text{TCFM CF}} \times 100$$

All quantitations are rounded to two significant figures.

1,1,1,2-tetrachloroethane	8-184[a]	8-184

Compound	8010 % recovery range	Lab % recovery range
bromoform	13-159	10-170
1,1,2,2-tetrachloroethane	8-184	5-190
1,2,3-trichloropropane	60-140[a]	60-140
bromobenzene	60-140[a]	60-140
2-chlorotoluene	60-140[a]	60-140
4-chlorotoluene	60-140[a]	60-140
1,3-dichlorobenzene	7-187	5-190
1,4-dichlorobenzene	42-143	45-140
1,2-dichlorobenzene	D-208[b]	1-200

[a] Value taken from CompuChem's historically generated acceptance range.

[b] D = Detected

There is general agreement between the lab acceptance criteria ranges and the ranges from method 8010. The check standards will therefore be run after every 10 samples, and the acceptance criteria ranges will be from Table 3 in method 8010 with the exception of the non-method 601 compounds which will use our laboratory's acceptance range. When a check standard fails to meet the recovery criteria, an initial calibration must be performed. Failure of the check standard means analysis cannot continue until system maintenance is performed and the check standard passes criteria.

6.3 Blanks

Method 8000 specifies that one method blank be prepared per analytical batch (up to 20 samples). For method 8010, a blank will be run either once per day per instrument or once per analytical batch, whichever is more frequent. A method blank consists of laboratory pure water spiked with bromofluorobenzene (BFB) and trichlorofluoromethane (TCFM) at 5 µl per 5.0 ml of water. The method blank must not contain any target compounds above the reporting limit. Exceptions to this are common laboratory solvents (i.e., methylene chloride) which may be present in blanks at 5X the stated detection limit.

6.4 Matrix Spikes

As specified in method 8000 (paragraph 8.5), duplicate matrix spikes will be analyzed per analytical batch (up to 20 samples). If a spike fails the recovery criteria listed in the check standard acceptance ranges table above, then a

8.0 Method Validation Summary

The following table summarizes the results of the method validation study performed June 5-6, 1989. The reporting level has been set by the validation data. The study was performed following the EPA requirements set forth in the Federal Register (Vol. 49, No. 209, Appendix B to Part 136, pp.43430-43431), and CompuChem's QA Program Plan, Appendix A.

Compound	Average % Recovery	MDL (µg/l)	Report (µg/l)
chloromethane	69.8	0.495887	0.50
vinyl chloride	69.8	0.541896	0.55
bromomethane	78.3	0.440651	0.45
chloroethane	85.3	0.495887	0.50
1,1-dichloroethene	89.0	0.303800	0.35
methylene chloride	102.0	0.797334	1.0
t-1,2-dichloroethene	85.5	0.294521	0.30
1,1-dichloroethane	93.0	0.338500	0.35
chloroform	84.5	0.320404	0.35
bromochloromethane	78.5	0.220327	0.25
1,1,1-trichloroethane	86.0	0.303831	0.35
carbon tetrachloride	83.5	0.310310	0.35
1,2-dichloroethane	79.0	0.211100	0.25
trichloroethene	87.0	0.282100	0.30
1,2-dichloropropane	76.0	0.276400	0.30
bromodichloromethane	80.0	0.398949	0.40
dibromomethane	87.5	0.383698	0.40
2-chloroethyl vinyl ether	88.5	0.379526	0.40
c-1,3-dichloropropene	60.0	0.252318	0.30
t-1,3-dichloropropene	81.5	0.234330	0.25
1,1,2-trichloroethane	85.0	0.218514	0.25
tetrachloroethene	89.0	0.276400	0.30
dibromochloromethane	63.0	0.258549	0.30
1,2-dibromoethane	84.0	0.328980	0.35
chlorobenzene	84.0	0.303830	0.35
1,1,1,2-tetrachloroethane	85.5	0.344348	0.35
bromoform	88.5	0.478210	0.50
1,1,2,2-tetrachloroethane	88.5	0.399944	0.40
1,2,3-trichloropropane	93.5	0.310310	0.35
bromobenzene	71.4	0.817600	0.85
2-chlorotoluene	74.0	0.211104	0.25
4-chlorotoluene	89.0	0.303830	0.35
1,3-dichlorobenzene	109.0	0.182025	0.20
1,4-dichlorobenzene	112.0	0.198360	0.20
1,2-dichlorobenzene	111.0	0.265346	0.30

Standard Operating Procedure (SOP) Documentation Form

Standard Operating Procedures (SOPs) describe in detail how tasks are performed in specific areas. Because they are used for training as well as for legal documentation, it is important that SOPs reflect the most current practices of the laboratory or department. In turn, we must keep careful records of who wrote or revised SOPs, when they became effective, and when it is time for SOPs to be reviewed. This form must accompany all SOPs to help us record that information.

Please be sure that the shaded area of this form is completed before you give the new or revised SOP to Quality Assurance for approval.

- This is a (check one) new procedure ✓ revised procedure _____
- Procedure area, title, and SOP number: GC VOLATILE, SOP for analysis of purgeable Halogenated volatile organic compounds in water by Method 8010 — Effective Date: 6/6/89
- Procedure prepared by: Roy Sutton (UPDATED BY Kelly Evans 11/18/91) — Date: 6/6/89
- Procedure approved by: (*If the lab manager prepared the SOP, a qualified second party should sign*) Doug McCormack — Date: 11-18-91
- This procedure meets the requirements set forth in the following References for Approved Methods:
  ~~Methods for the Chemical analysis of water and wastewater, EPA-600/4-79-020 revised March 1983 and 600/4-79-017, revised March 1983.~~
  1) Test Methods for evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd edition, revised September 1986.
  2) U.S. EPA Federal Register Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, Final Rule and Proposed Rule, Vol. 49, Part 209
- Procedure approved by Quality Assurance Representative: Janet G. Livingston — Date: 12-6-91
- Procedure received by Technical Communications: Suzanne Fischer — Date: 12/4/91

NOTE: One year from the date Technical Communications received this SOP, lab managers are required to review lab practices and revise the SOP if necessary.

Annual Review Date: 12/4/92

→ 3) Portions of standard Operating Procedure for analysis of Purgeable aromatic compounds and Methyl-tert-butyl ether, prepared by Bill Degardino, March 1989.

APPENDIX A.3

COMPUCHEM LABORATORIES, INC.

CHAIN-OF-CUSTODY PROCEDURES

Chain-of-Custody

The basic components for maintaining sample chain-of-custody (C-O-C) are:

1) samples must be relinquished into the possession of an authorized laboratory staff member, or

2) samples must be within the authorized staff member's line-of-sight, or

3) samples must be locked in a secured storage area with restricted access.

Furthermore, any change of possession or custody must be documented on appropriate chain-of-custody forms. This documentation must include both the initials of the individual relinquishing the sample and those of the individual receiving the sample, as well as the date of the custody transfer.

CompuChem® accomplishes these objectives through an elaborate document control system. This system includes procedures for documentation of the receipt of the sample into the laboratory using preprinted, numbered chain-of-custody records (although many clients provide their own C-O-C records which suffice). These records include information about the individuals collecting the samples, the collection date, time and location, and the type of analyses required. CompuChem's clients are responsible for field chain-of-custody, sample collection, handling and shipping.

When the samples are received in the laboratory, the C-O-C documents are signed and dated by the Receiving Clerk. The samples are logged into the Computerized Laboratory Management System (CLMS), and assigned unique sample identification numbers. The samples are then relinquished to the possession of a Sample Custodian, who has sole access to the locked sample storage refrigerators. The CLMS schedules the appropriate analyses and tracks the progress of sample

processing throughout the laboratory. Samples do not remain outside refrigeration more than 2 hours from receipt.

The custody of each sample can be determined at any point in time by reviewing Scheduling Details within the CLMS. A "paper trail" also accompanies the movement of the sample (or extracts, aliquots or digestates created from the as-received sample) throughout the lab, serving to document internally all changes in custody.

The integrity of the samples within the laboratory is assured by the security of the facility itself. Building security is controlled by an electronic card entry system. The exterior doors and the doors of various controlled-access areas are equipped with card readers. Each memeber of the staff has an access-card, which must be prominently displayed on their person, that is coded only for those areas where their job functions require access. The system also maintains a record of the movements, or attempted movements, of the staff throughout the building. A computer printout of this record is audited by a member of the Quality Assurance Department for verification of card coding and card entry transactions.

When the analysis is complete, the final extracts (for extractable portions of the sample) are kept in a locked freezer (if required) under sole custody of the Sample Custodian.

A complete description of CompuChem's sample tracking procedures and additional chain-of-custody details can be found in the Production, Planning and Control SOPs.

APPENDIX A.4

COMPUCHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURES FOR PRODUCTION PLANNING AND CONTROL

RE[illegible] JUN 0 8 1989

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. (If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.)

* Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat and gloves). These items need to be worn at all times when in the marked areas (blue line).

* Inspect each sample container before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, inspect these items to make sure that they are intact and record this observation on the chain-of-custody form (see Example 1, at the end of this SOP). If the custody seals, tapes, or padlocks are broken, contact Customer Service (for commercial samples) or the Sample Mangement Office (for EPA samples) for permission to continue processing the sample.

* Each container is opened under the hood and checked for breakage. Check for the condition of the refrigerant (whether any ice remains or whether the cooling packs are solid) and obtain the temperature of a representative sample (liquid samples only) by immersing a clean thermometer in the sample. Record the temperature on the Sample Record (see Example 2, at the end of this SOP).

* The temperature and pH are recorded on the log sheet. See Ex. 1 and 2 lab notices if a variance occurs.

* Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

* The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

* When a CompuChem SampleSaver® is received, record this receipt (on the file card) in the CLMS and insert the file card (contained in the SampleSaver®) into the file-card storage box, according to the date received.

* Remove samples from the shipping container and compare the sample identification information on the sample bottles to the sample information on the traffic sheets, packing lists, and chain-of-custody form included in the container (see Examples 3A and 3B, at the end of this SOP). If discrepancies exist, note the problem on the chain-of-custody form and notify Customer Service (for commercial samples) or SMO (for EPA samples).

* Each water VOA is checked for air bubbles and headspace, and noted on the chain-of-custody form.

* On each complete and correct EPA Chain of Custody and Traffic Report the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

* On each complete and correct Commercial Chain-of-Custody the statement 'Received in Good Condition' is written or stamped, initialed and dated by the receiving individual.

  'Received in Good Condition' is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain of custody seals), the exceptions are noted on the

  appropriate documents, initialed and dated.

* The statement 'Received in Good Condition' does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

* Check incoming samples against SMO scheduled receipts (for EPA samples).

* Log the sample in on the Accessioning Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

* For EPA samples, enter the samples' account data into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, contact customer service to check for the existence of the order. Then complete the order in the CLMS, and complete the EPA Scheduling Log (Example 4, at the end of this SOP).

* Enter sample into sample receipt portion of CLMS in order to generate a CompuChem number for each sample. Fill in the CompuChem number on the accessioning log sheet (this completes the log sheet).

* A CompuChem label is generated in numerical sequence.

* Label samples with the CompuChem number by wrapping each sample bottle with its computer - generated CompuChem sample label. Sample labels are color coded, and are to be rotated with a different color every 2 week period by the Supervisor of Receiving or the Supervisor designee.

* Each log sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review each log sheet is stamped as reviewed and initialed and dated.

* Transfer the labelled samples to the secure, locked walk-in cooler facility.

* The CompuChem number is listed on the original Chain-of-Custody next to the associated client ID when possible.

* Access the Quiz portion of the CLMS to produce the worksheets for EPA sample analyses. For EPA samples the system will generate volatile, semi-volatile, and pesticide worksheets. For commercial and inorganic samples, pull the appropriate worksheets from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with customer service. Note the following destinations for the various worksheets:

  Pesticide/Herbicide Worksheets: GC Lab Volatiles that do not require compositing: GC/MS Lab Inorganics: Inorganics Preparation Lab Volatiles requiring compositing, all EPA volatiles, acid/base-neutrals (commercial), semi-volatiles (EPA), and commercial TCDD's: Production Planning and Control for scheduling.

* To produce EPA quality control worksheets for the QC samples associated with a batch of samples, access the EPA Water or EPA Solid programs of Quiz in the CLMS and enter the samples' CompuChem Numbers; copy these worksheets on green paper. To assemble commercial QC worksheets, pull the appropriate green fraction worksheets from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular batch. These are printed after the entry of sample numbers into the system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary a copy of the Chain-of-Custody Record.

* Assemble commercial file folders for Report Integration; include in the production sample's folder the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; in the green quality-control folder include the QC Sample Record, which also goes to Report Integration.

* Assemble EPA file folders for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-Of-Custody, original air-bill, a copy of the Log Sheet (also called Accessioning Report), a copy of the EPA scheduling Log (see Example 4A, at the end of this SOP), Custody Tags (if received) and a grey envelop contents sheet (See 4b). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (See Attachments 4c and 4d). The original EPA Scheduling Log is put in the EPA Book (kept in the Receiving area).

* If problems arise concerning received samples, contact Customer Service (for commercial samples) or the Technical Management Staff (for EPA samples).

Attachment # 1

LABORATORY NOTICE

CompuChem # _ _ _ _ _ _ _ _ _

Sample ID _ _ _ _ _ _ _ _ _

Case # _ _ _ _ _ _ _ _ _

Type of Analysis _ _ _ _ _ _ _ _ _

Receipt Date _ _ _ _ _ _ _ _ _

The pH reading for the sample listed above was _ _ _ _ _, the required pH level is _ _ _ _.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

_ _ _ Preserve In-House by Inorganics Prep Lab

_ _ _ Analyze As Received, and Qualify with this Notice

_ _ _ Dispose - Client will resample

_ _ _ Subcontract Lab to Preserve

Supervisor Signature _ _ _ _ _ _ _ _ _ _ _ _

Date _ _ _ _ _ _ _ _ _ _ _ _

Attachment #2

LABORATORY NOTICE

CompuChem # _ _ _ _ _ _ _ _ _ _

Sample ID _ _ _ _ _ _ _ _ _ _

Case # _ _ _ _ _ _ _ _ _ _

Sample Type _ _ _ _ _ _ _ _ _ _

Receipt Date _ _ _ _ _ _ _ _ _ _

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C(+/-2C). The temperature of the sample listed above was _ _ _ _ .

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

_ _ _ Analyze As Received, and Qualify with this Notice

_ _ _ Dispose - Client will resample

Supervisor Signature _ _ _ _ _ _ _ _ _ _ _ _

Date _ _ _ _ _ _ _ _ _ _ _ _

CHAIN OF CUSTODY RECORD

PROJ. NO. | PROJECT NAME

SAMPLERS: (Signature)

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS							REMARKS

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	

Distribution: Original Accompanies Shipment; Copy to Field Files

Example 1

Example 2

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:
Turnaround: Priority: Due:
Analysis Codes:

Lab Instructions

Receiving Data

Sample Identification: SS Code:
SS Number: Receiving Codes:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_____	____	( )
_____	____	( )
_____	____	( )
_____	____	( )

Company Name:

Example 2 (Cont.)

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

COMPUCHEM LABORATORIES

EPA/CASE RECEIVING Sheet for: _____ Sheet 1 of 1

[ ] ORGANIC
[ ] INORGANIC

Case #: _____ Order #: _____

Case ID: _____ Tags: [YES] [NO] _____

Account #: _____ Region #: _____ TAT: _____ Chain of Custody: [YES] [NO] _____

Delivered By: _____ Freight Bill #: _____

	COMMENTS/REMARKS	SAMPLE ID	CC #	SD	ANALYSIS CODE	MATRIX	VOLUME	RQ	C/pH
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
12									
13									
15									
16									
17									
18									
19									
20									

Logged In By: _____
(Signature)

Reviewed By:

Received By: _____
(Signature)

COMPUCHEM LABORATORIES

Date Samples Received at Laboratory _____ SHEET ___ OF ___

Client _____ Order # _____ Chain of Custody (Yes) or (no) ___

Case ID: _____ TAT _____ SSe's: ___ ___ ___ ___ ___ ___ ___ ___ ___

Delivered By: _____ Freight Bill # _____ Subcontract # _____

#	COMMENTS REMARKS	REC	SAMPLE ID	CC #	SD	pH	Temp	Analysis Code	Matrix	Volume
01										
02										
03										
04										
05										
06										
07										
08										
09										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										

Logged In By: _____
(Signature)

Reviewed By:

Received By: _____
(Signature)

Example 3A

COMPUCHEM LABORATORIES

date shipped to consignee: _____ date received from consignee: _____

number of sample: _____

consignee name: _____

address: _____

_____

_____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

Example 3B

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

Example 4A

EPA SCHEDULING LOG FOR WEEK ENDING SATURDAY _____
(DATE)

CASE NUMBER: _____ NEW CASE: _____ OLD CASE: _____ COMPLETED CASE _____
REGION: _____ ESTIMATED SHIPPING DATE: _____
DELIVERABLES CODE: _____ TURN-AROUND-TIME: _____
ACCOUNT #: _____ CONTRACT #: _____ PRICE CODE: _____

QUANTITY EXPECTED: _____: WATERS ORDER #: _____
_____: SEDIMENTS/SOILS
_____: EXTRACTS
_____: DIOXINS AIRBILL #: _____

CONCENTRATION: _____: LOW _____: MEDIUM _____: HIGH

DATE SHIPMENT RECEIVED: _____ TEMPERATURE: _____
QUANTITY RECEIVED: _____: WATERS
_____: SEDIMENTS/SOILS TAGS: YES/NO
_____: EXTRACTS _____
_____: DIOXINS

CONCENTRATION: _____: LOW _____: MEDIUM _____: HIGH

PROBLEMS/COMMENTS: _____

_____

_____

_____

_____

SMO CONTACTED AT (TIME): _____ SPOKE TO: _____
RESOLUTION FROM SMO: _____

_____

_____

_____

EXAMPLE 4B

DATE:_____

CONTENTS FOR GRAY ENVELOPES

EPA: [ ] ORGANIC CASE
[ ] INORGANIC CASE
[ ] DIOXIN CASE

COMMERCIAL: [ ]_____
CLIENT

CASE#: _____ ORDER#: _____

NO. OF TRAFFIC REPORTS: _____

	YES	NO	N/A	ITEM	COMMENTS
1.	[ ]	[ ]		CHAIN-OF-CUSTODY	
2.	[ ]	[ ]		AIRBILL	
3.	[ ]	[ ]		DAILY LOG SHEET	
4.	[ ]	[ ]	[ ]	TRAFFIC REPORT (EPA)	
5.	[ ]	[ ]	[ ]	SHIPMENT RECORD (EPA)	
6.	[ ]	[ ]	[ ]	TAGS	
7.	[ ]	[ ]	[ ]	DAILY pH CHECK SHEET	
8.	[ ]	[ ]	[ ]	SMO COVER LETTER	
9.	[ ]	[ ]	[ ]	EPA SCHEDULING LOG	
10.	[ ]	[ ]	[ ]	COMMERCIAL SCHED. LOG	
11.	[ ]	[ ]	[ ]	SIS (#____-____)	
12.	[ ]	[ ]	[ ]	_____	

COMMENTS:_____

	SIGNATURES/INITALS	DATE
SAMPLE RECEIPT/SCHEDULING:		
QA SECTION (IF REQUIRED):		
DOCUMENTATION CONTROL:		

DATE:_____

Dear Linda,

Enclosed are the SMO and/or Regional copies of the Organic Traffic Reports (OTR) for Case #_____, received by CompuChem Laboratories on _____.

If you should have any problems or question concerning this package, please do not hesitate to contact Richard Bloom at extension #215, or myself at #219/220.

Thank you,

_____

Natalie Carter

Additional Comments:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SDG) TRAFFIC REPORT (TR) COVER SHEET

Lab Name: _____ Contract No.: 68-01-_____

Lab Code: _____ Case No.: _____ SAS No.: _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: _____ Sample Receipt Date: _____
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____

Sample Custodian Date

APPENDIX A.5

COMPUCHEM LABORATORIES, INC.

DATA PROCESSING PROCEDURES

8.0 Data Processing

This section summarizes the manner in which all aspects of data proccessing are managed and evaluated in order to maintain data integrity and characterize data quality. These processes include data collection, validation, storage, transfer, and reduction. Specific details of the procedures used by the automated data processing and computer systems operations are documented in the individual laboratory SOPs and the Report Preparation Department SOPs.

8.1 Collection

Analytical data are generated from the GC/MS computer software, GC computer, ICP computer, Atomic Absorption Spectrophotometers, Autoanalyzers, and associated laboratory instrumentation. The outputs include identifications of compounds or elements, concentrations, retention times, and comparisons to standards. Outputs are in graphic form (chromatograms), bar graph (spectra) and printed tabular form. The outputs are in standard format specified for each analysis type and are monitored for consistency. If incomplete or incorrect output is generated, corrective actions are taken according to SOPs established for each type of analysis and consistent with the manufacturer's recommendations.

All outputs of each of the instruments may be checked manually for each procedure (e.g., GC chromatographic peak area integration and calculations may be reviewed manually for baseline designation and quantitation). In the data review process (see section 8.2, Validation), the data produced are compared to information concerning the sample processing history, sample preparations, sample analysis, associated QC data, etc. to evaluate the validity of the results.

Ancillary data produced for internal records and which may not be required by the customers as part of the analytical data package include the following: laboratory worksheets, laboratory notebooks, sample tracking system forms, instrument logs, calibration records, maintenance records, standard prepartion records, and associated quality control sample data. These data are available for inspection during audits to verify the validity of data and are also deliverable, depending on the client's needs.

A complete record of each sample's history is available for documenting its progress through the laboratory from sample receipt to reporting. Document control (see section 4.4 of the QA Plan) and chain-of-custody (see Appendix E) requirements present additional information describing these documentation and archiving processes.

8.2 Validation

Data validation takes on two scales. First, the Quality Assurance Department is charged with the responsibility of monitoring all laboratory QC activities, and to verify that systems are in control. These responsibilities and the manner in which they are executed are described in the QA SOPs as well as this QA Plan. The QP Department therefore plays a role in data validation in the context of the overall QA Program.

Of course, data validation also occurs on a sample-by-sample basis. This is the responsibility of the various levels of data review taking place within the laboratory. The first level of review occurs "at the bench"--this is the initial review by the instrument operator or analyst, responsible for assessing the following:

- cross-checking all sample identification numbers on worksheets, extract vials/digestate bottles, and instrument outputs
- calculation of surrogate recoveries and internal standard responses (when applicable), and verification that QC acceptance criteria are met.
- verification that all calibration, tuning, linearity, and retention time drift checks are within QC acceptance criteria
- verification that all target analytes are within the instrument's analytical range and deciding on appropriate dilutions when necessary
- determination that peak chromatography and other instrument performance characteristics are acceptable
- verification that chain-of-custody is intact based on accompanying paperwork

The second level of review is performed by the in-lab Data Review staff. In the GC/MS Laboratory, these reviewers are all experienced Mass Spectroscopists qualified to perform mass spectral interpretation. GC Lab and Inorganics Lab Data Reviewers are degreed, senior-level chemists. The Senior Data Reviewer,

Manager of Data Review or Lab Manager also audit a percentage of these data prior to being released to the Report Preparation Department. In-Lab Data Reviewers verify all assessments previously made by the operator/analyst, and also evaluate the following:

- verification that all quality control blanks meet QC requirements for contamination, and that associated sample data are appropriately qualified when necessary
- calculation of matrix spike recoveries and duplicate RPDs, and verification that accuracy and precision QC criteria are met
- comparison of all injections of a sample, and comparison of matrix spikes with the original unspiked sample, for acceptable replication
- qualitative identification of all target analytes using specific SOP interpretation criteria
- verification of computer quantitation of all target analytes, including evaluation of Extracted Ion Current Profiles (EICPs) and of chromatograms for proper resolution and integration, when necessary
- verification that analytical worksheets have been completed by the operator/analyst, including date and initials
- for pesticide GC/MS or GC confirmation analyses, verification that target analytes were within retention time windows and/or evaluation of spectra for proper identification, and comparison to initial analysis
- for GC/MS analyses, evaluation of Library Search mass spectra, characterization of tentatively identified compounds, and verification of calculations for estimated concentrations of these compounds
- verification that GLP was followed relative to the correct procedure in making changes to data

The completed data package, which has been reviewed on an analytical fraction basis (i.e., volatiles, acids, base/neutrals, pesticides), is then forwarded to the Report Preparation Department. The package is then integrated with other fractions from the same sample, and with associated deliverable items as required by the client, and forwarded to the Final Technical Review staff for

the third level of review. The Final Technical Reviewer, also a senior chemist and experienced data validation specialist, assesses the complete data report (or "case", for CLP-format reports) and double-checks all items previously validated by the in-lab Data Reviewer. Additional assessments include the following:

- review of all data summary documents and verification of correct transcription from raw data
- comparative evaluation of data from individual fractions of a sample, and of samples from the same site, project or case, for consistency of analytical results and resolution of discrepancies
- checks data report or case for completeness
- for CLP-format reports, a "case narrative" must be written that authorizes release of the data, provides end-users a "history" of the sample processing, documents the quality control process used and exceptions to Statement-of-Work criteria, and summarizes any corrective actions taken.

Upon completion of all levels of review and authorization of data release by the Final Technical Reviewer, the data report (or case of reports) is sent to the Deliverables Department for mailing.

Senior members of the Quality Assurance Department are also required to audit approximately 10% of all analytical data. The QA auditor performs the same assessments as the Final Technical Reviewer. Findings from these data audits are presented in a report to management, as described in section 10.0 of the QA Plan.

8.3 Storage

At every stage of data processing at which a permanent collection of data is stored, procedures are established to ensure data integrity and security. Specific QA Project Plans indicate how specific types of data are stored with respect to media, conditions, locations, retention time, access. The following chart indicates general guidelines as detailed in Production, Planning Control SOP 2.9:

Media	Conditions	Location	Retention Time	Access
Hardcopy	locked warehouse	off-site	client-specific	Document Custodian or other designated personnel
Magnetic Tape	locked warehouse (environment controlled)	off-site	indefinitely	Document Custodian or other designated personnel

8.4 Transfer

All data transcriptions for final reports to clients are perfomed by Report Preparation Clerks. For non-CLP reports, the reportable data is reviewed and approved by the Final Technical Reviewer, then word-processed by computer. Validation of the word-processing function is performed by a proofreader prior to release of the data. For CLP reports (whether to EPA or commercial clients), all raw data are reduced into deliverable format by Report Preparation Clerks, including the summary of data onto forms required by the Statement-of-Work. Data summaries are accomplished by utilizing a PC-based software system that extracts data directly from the laboratories' computers. The data are then sent to the Final Technical Reviewer for validation. In either case, the Final Technical Reviewer is provided with both the deliverable report and the non-deliverable back-up data, and must validate all transcription processes.

8.5 Data Reduction

Data reduction includes all processes that change either the instrument/computer-generated values, quantity of data values or numbers of data items, and frequently includes computation of summary statistics. Documentation of the calculation process is required. In most cases, a programmable calculator, PC spreadsheet or computer program is used in this process. The documentation allows the reviewer to verify the validity of the data reduction process. All computer-generated compound lists containing the reportable results include formulae used in performing the calculations.

CompuChem's policies regarding the use of significant figures and rounding of results are outlined in Appendix H. An extra significant figure is carried through all calculations until the final, reportable result is generated. Analytical results are never corrected for blank (background) contamination, but are flagged and footnoted appropriately.

APPENDIX A.6

COMPUCHEM LABORATORIES, INC.

CORRECTIVE ACTION AND PERFORMANCE AUDITS PROCEDURES

10.0 Corrective Action

10.1 Introduction

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because routine data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiences or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

CORRECTIVE ACTION REPORT

DATE: _____

PROBLEM / DEFICIENCY:

_____

_____

_____

_____

_____

IDENTIFIED BY: _____

REFERRED TO: _____ (QA)

CORRECTIVE ACTION TO BE TAKEN: TARGET DATE: _____

_____

_____

_____

_____

_____

_____

FOLLOW-UP AUDIT FINDINGS:

_____

_____

_____

_____

RESOLVED? _____ DATE: _____

SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [ ] NO [ ] TARGET DATE: _____

This form to be filed with the Quality Assurance Clerk for permanent record.

10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- -- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures. Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

- -- Summary of any changes in certification/accreditation status
- -- Involvements in resolution of quality issues with clients or agencies
- -- QA organizational changes
- -- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)
- -- Training and safety issues, if not already covered in audit reports during the period
- -- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)
- -- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

10.4 Laboratory Performance Reports

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of an in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem®, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem® also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem® also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

1. The quality control, including necessary corrective actions , are being applied
2. Adequate facilities and equipment are available to perform the client's required scope-of-work
3. The personnel are qualified to perform the assigned tasks
4. Complete documentation is available, including sample chain-of-custody

5. Proper analytical methodology is being applied

6. Acceptable data handling techniques are being used

7. Corrective actions identified in any previous on-site visits have been implemented, and

8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory Evaluation Checklist. In response to System Audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's Standard Operating Procedures.

QUALITY ASSURANCE AUDIT SUMMARY

		REJECT LOCATION/CAUSE							SUMMARY		
SAMPLE NUMBER	FRACTION							COMMENTS	corr* failure	crit** failure	status

Missing/Incorrect:

CAM/I	Calculations missing/incorrect
CCM/I	Condition code missing/incorrect
DFM/I	Data footnote missing/incorrect
CFM/I	Correction factor missing/incorrect
DWI	Dry weight/percent moisture incorrect
FNI	Filename incorrect
FFM/I	Form 4 missing/incorrect
LSM/I	Library search missing/incorrect
QNM/I	QA Notice missing/incorrect
RRM/I	Reportable run missing/incorrect
SPM/I	Spectrum missing/incorrect
SRM/I	Sample receiving information missing/incorrect
SSM/I	Surrogate Summary Form missing/incorrect
STM/I	Standard package missing/incorrect
TFM/I	Tuning Form missing/incorrect
UNM/I	Units missing/incorrect
WSM/I	Worksheet missing/incorrect/incomplete
OAM/I	OADS missing/incorrect/incomplete

Qualitative/Quantitative Errors:

HNR	Hit not reported, but should have been
HRE	Hit reported in error, should not have been reported
HAI	Hit amount reported incorrectly
CFN	Correction factor not applied to hit
SFI	Significant figures (or rounding off) incorrect
TRE	Transcription error

Miscellaneous Errors:

ISF	Internal standard area monitor indicates failure
ODI	OWA date or time incorrect
RNL	RIC not labeled
SOL	Surrogate(s) actually outside limits
WOU	Whiteout used on documents (deliverables)
NSO	Not signed off
CNI	Change not initialed

Condition Code Applications:

CS	Carryover suspected
CT	Contamination evident
RU	Repeated unnecessarily
SF	Spikes failed
UN	Unacceptable, not needed

APPENDIX A.7

COMPUCHEM LABORATORIES, INC.

FACILITIES, EQUIPMENT, AND SERVICES

6.0 Facilities, Equipment and Services

6.1 Introduction

This section describes the facilities at CompuChem®, the instrumentation and peripheral equipment, and the services provided in maintaining the facility.

CompuChem® is located in Research Triangle Park, NC, 15 miles west of Raleigh. The total facility is comprised of both the Environmental and Forensic Drug Testing Operations of CompuChem® Laboratories, Inc. The two operations have separate laboratories that function independently, including separate computer systems. Much of the office space is also separate, however, many administrative functions overlap (i.e., Accounting, Quality Assurance, Human Resources, Computer Operations) and share common office space. Facility space allocation is described in section 6.2, and includes the Environmental Operations laboratory space, Environmental office space, and administrative office space common to both operations, totaling approximately 64,000 square feet. The two operations share two adjacent buildings which are connected by a permanent, enclosed walkway. Electrical power is supplied by Duke Power Company, with a service capacity of 2000 amperes at 480 volts. The enviornmental controls for the heating, ventilating, and air conditioning systems are Honeywell Electric and provide automatic starting and stopping as well as temperature control. All critical temperature areas such as refrigerators, freezers and computer rooms are monitored 24 hours/day by an off-site monitoring firm. The temperature of the refrigerators and freezers is maintained by a standby generator in the event of a power failure. The electrical power to the computer room is regulated by a power conditioner.

Building security is maintained at all times. The main entrance is monitored by a full-time, contracted security staff (24 hours/day, seven days/week). Visitors must sign-in at the security guard's desk and be escorted through the facility by members of the staff. The exterior doors as well as the doors of various controlled access areas within the building are equipped with electronic card readers, controlled by Rusco Electronic Card Entry Access System. A burglar alarm system has been integrated with the Rusco system to provide protection when the facility is closed. Smoke detectors, as well as associated pull stations and fire alarm horns, are provided throughout the building for fire protection. Adequate fire extinguishers and emergency equipment are also provided. The fire burglar alarms are also monitored by the off-site security firm. When an alarm sounds, the off-site personnel alert the appropriate laboratory personnel, the Sheriff's office, or the Fire Department, as necessary.

CompuChem® Laboratories contains sophisticated, state-of-the-art instrumentation and data processing equipment capable of performing most organic and inorganic analyses. Two Hewlett Packard-3000 Series 70 mainframe computers are dedicated to scheduling and tracking sample analyses through the laboratories and are directly networked to GC/MS instrumentation. An HP-3000 Series 950 mainframe provides system redundancy in the event of primary system failure, and handles additional production coordination. One of two HP-3000 Series 39 microcomputers is dedicated to systems research; the second handles all accounting functions. The Computerized Laboratory Management System (CLMS) is accessed by laboratory, marketing, systems, and accounting personnel via more than 90 CRT computer terminals.

The Manager of Facilities and Safety, Manager of Instrumentation, and Manager of Computer Operations are primarily responsible for the evaluation, selection and maintenance of all facilities, instrumentation, and computer equipment, respectively. The Manager of Facilities and Safety is also responsible for overseeing general housekeeping services and functions as the Laboratory Safety Officer. In this capacity, the Safety Officer conducts periodic safety inspections and manages the activities of the Safety Committee.

All analytical instruments are maintained by a staff of full-time service technicians, operating during all three shifts, seven days/week (also available on-call on weekends). Instrument log books are maintained for each individual instrument in each of the laboratories (GC/MS GC, Inorganics), for recording routine maintenance performed by the operator or laboratory staff. Additionally, service records for each instrument are kept in the Maintenance Department to record all routine and non-routine maintenance performed by service technicians.

The Pure Water Room houses a state-of-the-art water purification system. Municipal water is fed through two mixed-bed ion exchange cylinders and a high capacity activated carbon tank. The effluent is pre-polished by two mixed-bed ion exchange columns, distilled in a Corning 12-liter all-glass still, then passes through a Megapure Polishing System. This final purification process feeds water through two more mixed-bed ion exchange cartridges, and activated carbon column and a clarifying filter. Water quality is monitored daily by an in-line specific conductivity meter, and by the various method blank and instrument blank QC checks performed on the water. A similar system is used at

an off-site warehouse facility to produce pure water used in the trip blanks that accompany SampleSavers (sample coolers) into the field during sampling operations. The Sample Preparation Laboratory and QA SOPs include additional information regarding the operation of the stills.

Two other laboratories have systems in-place to perform additional processing of the water from the Pure Water Room. Teflon transfer lines feed the water into the Inorganics Sample Preparation Laboratory and Volatile GC/MS Laboratory systems. Inorganics Lab pure water passes through an additional Millipore Pure Water System (with ion-exchange cartridges and a carbon filter), and water for the Volatile Lab is sparged with nitrogen in an all-glass reservoir for 24 hours prior to use.

The laboratory also has a full complement of support equipment and instrumentation, such as glove boxes and hoods, walk-in refrigerators, freezer units, autoanalyzers, and sonicators.

The following sections describe the laboratory area by function and equipment. The floor plan was designed to allow for the efficient and secure movement of samples and data between work areas.

6.2 Laboratory Areas

Shipping and Receiving: This area is located adjacent to the laboratory section of the building. Samples arriving are identified and introduced into the scheduling and control system. The sample receiving area for environmental samples has about 1,570 square feet of floor space. The receiving area has 102 square feet of bench space for receiving and opening samples, three data entry stations, one laboratory sink and ample storage shelving.

Walk-in Refrigeration System: This area is accessed from the Sample Custodian's area outside of Sample Receiving. This 2,500 cubic feet system has two independent refrigeration units, is temperature controlled to 4°C ± 2°C and is equipped with an activated carbon air filtering system, which maintains an environment free of organic vapors. The temperature is recorded daily. Both entrances are secured by locks and the temperature-activated alarm system is tied into a private security service. In the event of unauthorized access or temperature fluctuations, appropriate parties are notifed by the private security service. A generator maintains the temperature in the event of a power failure.

Organic Extractions and Inorganic Preparations Laboratory: This area is equipped with hoods as well as extraction equipment sufficient to process many thousands of samples per month. The environmental sample preparation laboratory has 2,008 square feet of space, four 8' fume hoods, three IEC centrifuges, two vacuum ovens, two sinks, six water baths, and 220 square feet of bench space. The air handling system for the sample preparation laboratory was custom designed for the extraction process. Conditioned 100% outdoor air is supplied

into the room through linear diffusors and vented through exhaust ducts which extend from wall-to-wall on the north and south ends of the laboratory. This method maintains air flow at the work/stations at all times and virtually makes the room a large walk-in fume hood. A complete air exchange occurs every two minutes. Separate exhausts are provided for furnaces and hoods. Adequate cabinet space is provided. Specially-designed water baths controlled and programmable to temperature and duration are also used. The glassware preparation room has 750 square feet of floor space and is equipped with two glassware washers, 26 feet of stainless steel counters with four built-in sinks, and one 72 cubic foot annealing oven.

Solvent Storage Area: This area is accessible through a secured door adjacent to the extraction and preparation area. The room is designed with reinforced concrete walls, an automatic halon fire-extinguishing system, alarm systems and a roof that relieves pressure in the event of an accident.

GC Laboratory: The laboratory's nineteen gas chromatographs are equipped with autosamplers or purge-and-trap devices (Tekmar LSC-2) and are interfaced with a Hewlett-Packard 1000 laboratory computer for data processing (all of which are installed on a raised computer floor). A variety of detectors are attached to the GCs, including Flame Ionization (FID), Flame Photometric, Electron Capture, Thermionic Specific (also called NPD or AFID), Photoionization (PID), and Electrocoulometric (Hall) detectors.

GC/MS Laboratory: The special features included in this area are numerous. All twenty-three GC/MS systems are raised on a computer floor. This allows

gas, water, cooling and exhaust systems required to support each instrument to be introduced to the room independently, beneath the floor. There are 12 distillation units for cyanide and 8 units for phenols distillation.

Equipment is arranged in efficient work stations. In this way, specific instruments can be utilized for specific types of analyses. Several instruments are physically isolated from the rest of the GC/MS Laboratory by a glass wall (with an independent air handling system) dedicated to volatile organic analyses. These instruments are never subjected to semi-volatile work; therefore, cross-contamination of the instruments is eliminated. Furthermore, each station of instruments is staffed by experts familiar with the procedures associated with each specific method. This staffing system allows intimate daily interaction between the operator, his/her instruments and the methodologies required. All other instruments are dedicated in a similar fashion. The GC/MS Laboratory has a total of 3,380 square feet of space, and is provided with an individual power supply from a breaker panel located within the lab. The GC/MS instruments are powered by three 1-phase, 75 KVA 480/220 volt isolation transformers. Helium, the carrier gas used, is supplied from a manifold system in an adjacent room through a piping system under the raised floor. There are three of these systems, each having a catalytic scrubber to remove traces of oxygen and water, prior to entering an instrument.

The 23 GC/MS instruments are configured with both packed and capillary GC columns, and have accessories for purge and trap, direct injection, or solid probe for introduction of samples. Both electron impact and chemical ionization sources are available. Each GC/MS instrument is equipped with its own dedicated microprocessor for data processing.

Standards Laboratory: This area is separated completely from all other laboratories and is equipped with its own GC instrument. Refrigeration, glove box and hood units are located in this area. The entrance to this area is locked at all times and secured by a cypher lock.

Inorganics Laboratory: This area is separated completely from all other laboratories and has Inductively Coupled Plasma (ICP), Technicon autoanalyzer, Atomic Absorption Spectrophotometers (AAS) and UV/visible spectrophotometer systems. Several other analytical instruments required to perform classical analyses are also located in this laboratory. Hood systems are also an integral part of this laboratory.

Mercury is detected by flameless-cold-vapor methods established by the USEPA (Cold Vapor Technique). For maximum data management, the Inorganics Laboratory uses a mini-computer (Digital, PDP11/73) interfaced to the ICP instrument (Jarrel Ash, Model 1100).

Extract Storage: Sample extracts are stored in specially-designed refrigeration units located adjacent to the Walk-in Refrigeration System. These refrigeration units are kept locked and may be accessed only by a sample custodian. These refrigeration units are also connected to an alarm system. In the event of temperature fluctuations outside acceptable levels (4°C $\pm$ 2°C), appropriate parties are notified by a private security service and the problem is corrected by laboratory staff.

High Hazard Laboratory: A limited access laboratory has been designed for sample preparation aspects associated with high-hazard samples. For example, all samples requiring analysis for 2,3,7,8-TCDD are prepared in this lab. Access to the laboratory is by means of a cypher lock. The hoods are equipped with an HEPA filtration unit. Laboratory personnel use more sophisticated protective clothing than other extraction laboratory personnel (i.e. full sack suits, booties, face masks, etc).

FACILITY SPACE ALLOCATION

TOTAL LABORATORY BUILDING SQUARE FEET 24,005

1.	Sample Receiving	1,570 sq. ft.
2.	Glassware Prep	750 sq. ft.
3.	Organic Extractions and Inorganics Sample Preparation	2,008 sq. ft.
4.	High Hazard Lab	450 sq. ft.
5.	GC/MS	2,840 sq. ft.
6.	Computer Room	1,450 sq. ft.
7.	Standards Laboratory	312 sq. ft.
8.	Metals (Inorganics) Instrumentation Lab	650 sq. ft.
9.	GC Lab	1,200 sq. ft.
10.	Solvent Storage	542 sq. ft.
11.	Utility	960 sq. ft.
12.	Walk-In Refrigeration System (2 units)	250 sq. ft.
13.	Miscellaneous (Canteen, Corridors, Rest Rooms, etc.)	5,000 sq. ft.
14.	Office*	6,023 sq. ft.

TOTAL PAMLICO BUILDING SQUARE FEET 55,487

1.	Office*	40,142 sq. ft.

TOTAL COMPUCHEM LABORATORIES, INC. FACILITIES RESEARCH TRIANGLE PARK, NC* 79,492 sq. ft.

* includes both Environmental and Forensic Drug Testing Operations.

GAS CHROMATOGRAPH LABORATORY EQUIPMENT

Item	Model#	Serial#	CChem#	A-D#	Type	Installed
GC	VARIAN 3700	58760308-13	000000	2&3	DUAL ECD AUTOSAMPLER	1980
GC	VARIAN 3700	71280469-13		7&1	DUAL ECD AUTOSAMPLER	1980
GC	VARIAN 3700	32968966-11			FID NPD	1980
GC	VARIAN 3700	74550509-13		23	FID	1982
GC	HP 5880	2236A04163		21	FID	1982
GC	VARIAN 3400	2006	001177	5	FPD	1986
GC	VARIAN 3400	2310	001175	0	ECD NPD AUTOSAMPLER	1986
GC	VARIAN 3400	2309	001178	4	ECD NPD AUTOSAMPLER	1986
GC	VARIAN 3400	2312	001173	6	ECD FID AUTOSAMPLER	1986
GC	VARIAN 3400	3623		9	ECD FID AUTOSAMPLER	1986
GC	VARIAN 3400	3052		10	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2308		12	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2307	001174	14	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	2311	001179	24	ECD AUTOSAMPLER	1986
GC	VARIAN 3400	3053	001357	19	HALL DET	1985
	TEKMAR LSC-2	144			PURGE AND TRAP	
	TEKMAR ALS	1016	001647		AUTOSAMPLER	
	O.I. 442					
GC	VARIAN 3400	3054	001356	20	PID DET	1985
	O.I. 4460	171-6-9B	001499		PURGE AND TRAP	
	HNU PI-52	620045	001362			
GC	VARIAN 3400	2306	001176	18	PID	1985
	TEKMAR LSC-2	1821	001241		PURGE AND TRAP	
	TEKMAR ALS	1041	001648		AUTOSAMPLER	
	HNU PI-52	620100				
GC	VARIAN 3400	2005	000953	17	HALL	1985
	TEKMAR LSC-2	1556	001316		PURGE AND TRAP	
	TEKMAR ALS	902	001649		AUTOSAMPLER	
	O.I. 4420	6644-5-102				
GC	VARIAN 3400	3055	001358	16	PID	1985
	O.I. 4460	521-6051C	001507		PURGE AND TRAP	
	O.I.		001508		LOOP SAMPLING MODULE	
	O.I.	365-6-0020	001509			
	HNU PI-52	05836				
OVEN	BLUE M SW-11TA-1	SW365	001353		OVEN	
COMPUTER	HP 1000				ALS SYSTEM DATA PROCESSING	

CHARCOAL AIR FILTERING SYSTEM

GC/MS LABORATORY EQUIPMENT (ENVIRONMENTAL)

OWA #	Serial#	Type Of Application	Installed
OWA - 1020	12137-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12391-3-0281	CAPILLARY COLUMN	9/81
OWA - 1020	12141-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12138-0980	CAPILLARY COLUMN	9/81
OWA - 1020	12140-0980	CAPILLARY COLUMN	9/81
OWA - 1020	11957-2-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-3-0180	CAPILLARY COLUMN	9/81
OWA - 1020	11957-4-0180	CAPILLARY COLUMN	9/81
OWA - 1020		VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	11957-1279	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-2-0280	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391 0281	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12139-0980	VOA-LSC/PURGE AND TRAP	9/81
OWA - 1020	12391-1-0380	VOA-LSC/PURGE AND TRAP	6/82
OWA - 1020	12391-4-0381	CAPILLARY COLUMN	9/81
OWA - 1020	12391-5-0381	CAPILLARY COLUMN	6/83
OWA - 1020	12645-1-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-4-1181	VOA-LSC/PURGE AND TRAP	6/83
OWA - 1020	12645-6-1281	CAPILLARY COLUMN	6/83
OWA - 1020	12645-3-1181	CAPILLARY COLUMN	6/83
OWA - 1020	12645-2-1181	CAPILLARY COLUMN	6/83
OWA - 1020	S12645-5-1281	VOA-LSC/PURGE AND TRAP	6/83
INCOS 50	13954-0387	HP-GC WITH CAPILLARY COLUMN	1987

INORGANIC LABORATORY EQUIPMENT

Item	Make	Model#	Serial#	Installed
AUTO ANALYZER II	TECHNICON	TRAACS 800		1987
CIRCULATING BATH	PRECISION			1987
ANALYTICAL BALANCE	METTLER	MODEL HL 52	A76373	1980
ICP	JARRELL ASH	MODEL 1100	22483	1986
MICROPROCESSOR IONALYZER PH METER	ORION	ORION 901	93353	1979
UV VISIBLE SPECTROPHOTOMETER	VARIAN CARY	219	0438812	1981
CYANIDE/PHENOLS AUTOANALYZER	TECHNICON	AAII	GG0797940	1980
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2027	1987
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 22(857)	2127	1986
ATOMIC ABSORPTION SPECTROPHOTOMETER	INSTRUMENTATION LABORATORY	VIDEO 12(857)	2128	1986
VAPOR GENERATION ACCESSORY	AVA	440	1625	1986

6.4 Instrument Maintenance

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

EXAMPLE 1

COMPUCHEM SERVICE REPORT

R-1234 A

INSTRUMENT NO	1 2	DATE	0 7 2 3 8 6	TIME	1 1 5 1	OPERATOR: Susan Bass

GC oven not heating

START DATE	0 7 2 3 8 6	END DATE	0 7 2 3 8 6	RESPONSE TIME	5	S/E Lee Gregory
START TIME	1 0 1 5	END TIME	1 0 5 7	TOTAL DOWNTIME	1 2	JOB COMPLETE? YES X NO

ASSEMBLY NO.	REV LEVEL	REPAIR CODE	FAILURE CODE	COMPONENT DESIGNATOR	PART NO.	DESCRIPTION	QTY	COST
7 0 1 7 8 9 1 4 3 2 0 1	0 A	0 4	0 6	F 4	3 A 6 4 A	Fuse	1	1 35

COMMENTS: Oven not heating, 24 volt relay not engaging found fuse F4 blown in power supply. Replaced fuse F4, checked +24 volts, checked operation of GC, gave back to operator L.G.

OPERATOR ACCEPTANCE:	DATE 0 7 2 3 8 6	TIME 1 1 0 0	REV07298216

EXAMPLE 1 (CONTINUED)

INSTRUCTIONS

1. INSTRUMENT NO. - LIST AS 05 FOR OWA05, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.

2. DATE & TIME - ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.

3. OPERATOR - WHO YOU ARE.

4. PROBLEM CODE & DESCRIPTION - USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.

5. USE BLACK INK ONLY & WRITE OR PRINT LEGIBLY.

PROBLEM CODES (CAUSE & EFFECT)	
P.M.	000
CANNOT MEET TUNE	001
IDOS ERRORS - LIST AND FULLY DESCRIBE WHAT THE DATA SYSTEM WAS DOING	002
POOR SENSITIVITY	003
DRIFTING RET. TIMES	004
NO SPECTRA OR NO MS RESPONSE	005
SOFTWARE ANOMALIES	006
GAS CHROM.	007
DISC DRIVE	008
PRINTER	009
PURGE & TRAP	010
VACUUM FAULT	011
AIR LEAKS	012
TERMINAL	013
DATA SYSTEM	014
CANNOT BOOT	015
UNKNOWN	016

REPAIR ACTION CODES	
PIRATE PARTS	100
ADJUSTMENT - ELEC	102
ADJUSTMENT - MECH.	104
REPLACED ASSY.	106
RETURNED TO VENDOR REPAIR	108
RETURNED TO VENDOR WARRANTY	110
REQUESTED IN-HOUSE VENDOR SERVICE	112
WAITING FOR PARTS (NOTE P.O. #)	114
CLEANED SEPARATOR	116
CLEANED MASS FILTER	118
CLEANED SOURCE	120
REPLACE PART	122
REPAIR IN-HOUSE	124
UNABLE TO REPRODUCE	126

FAILURE ANALYSIS CODES	
UNKNOWN	200
MISCELLANEOUS	202
OPERATOR ERROR	204
SOFTWARE	206
HEADCRASH	208
MECH. DEFECT	210
OUT OF ADJUSTMENT	29*
INTERMITTANT	29*
EXCESSIVE NOISE	29*
EXCESSIVE WEAR	212
SHORTED COMPONENT	214
OPEN COMPONENT	216
FAULTY CRIMP	218
POOR CONTACT	220
POOR SOLDER JOINT	222
DIRTY/DUSTY	224
LEAKING	226
REPLACE * WITH	
1 = ELECTRICAL	
2 = MECHANICAL	
3 = VACUUM	
4 = SOFTWARE	

REV07/29821600

PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE*

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Sigma 3 GC								NOTE: Applicable procedures are presented in the Finnigan Operator Manual(s), unless otherwise specified.	
1. Line fuses for the GC	Inactive GC, blown fuse						X	replace fuse	
2. Injector for packed columns							X		
3. Splitless injection for capillary columns							X		
4. Injector septum in the GC	obstruction, leaks	X					X	clean, inspect or replace as required	
5. Carrier gas connections/couplings	leakage						X		
6. Carrier gas filter in the GC	replace when new gas cylinder is installed						X		
7. Filter, flow controller	dirty filter			X				replace filter clean	
8. Capillary column							X	inspect or replace as needed	
9. Packed column (glass type)	excessive usage, leaks at injection and interface port of the zone-heating block						X		
10. Packed column (metal type)							X		
11. Detector port to GC/MS interface							X		
12. GC cool down fan		X		X				inspect and/or replace	

*These maintenance procedures meet or exceed Finnigan's recommended preventive maintenance checks and services.

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL: daily	bi-monthly	3 months	6 months	yearly	as required	SERVICE INTERNAL	PROCEDURE
Mass Spectrometer									
1. Glass jet separator	obstruction or glass						X	clean or replace	
2. Glass jet separator ferrules	breakage						X	replace	
3. Mass analyzer head assembly (in the vacuum manifold)	gross leaks, presistent pressure due to degasing of trapped gases in the vacuum system						X	inspect	
*magnet well flange assy	leakage, faulty CAL gas pressure (see Pirani gauge)						X	inspect	
*CAL gas valve assy							X	inspect	
*vent valve assy							X	inspect	
*water flow sensor switch	faulty switch						X		
4. Quadrupole mass analyzer				X				inspect and/or replace	
5. Electron multiplier							X		
6. Alcatel vacuum pumps (2)				X				purge weekly and replace oil	
7. Pfeiffer turbo pump	dirty oil								
8. Balzer turbo pump				X					Balzer Manual pg...23
9. Vacuum system filter/drier	excessive use, dirty filter						X	clean & inspect	
10. Ion Source				X				replace	
*ion source filament assy	lack of sensitivity, faulty peak shape, no autotune			X				clean, inspect or replace as required	
*collector		Service concurrently with every filament assembly replacement							
*lens									
*aperture									
*ion volume									
GC/MS Interface Oven									
1. Capillary interface tubing	plugged						X	clean, inspect and/or replace	
2. Separator divert fitting				X			X		
3. Vacuum divert valve				X			X		
Power Module									
1. MS power supply				X			X	measure & verify PCB	
2. Turbo power supply				X			X		
Card Cage Module									
1. Air filter at bottom of cage	dirty filter, obstruction of air flow			X				clean and/or replace	
2. Fan	burned out fan			X			X		
3. Signal cable on Digital I/O PCB							X	inspect for secure fit	

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Nova Computer								Inspect and/or replace	
1. Fan	faulty fan rotation			X					
Perkin-Elmer Disk Drive									
1. Output signal				X				check and verify	
2. Adjustable DC voltages (+5V, +13V, -13V)				X					
3. Brushes								clean and/or replace	
4. Positioner carriage guide rails					X			clean and inspect	
5. Spindle chuck and cone					X			clean and inspect	P/ECEM Manual
6. Read-write heads				X				inspect and repair	P/ECEM Manual
7. Fixed disk				X					
8. Air filter						X			P/ECEM Manual
*prefilter				X				replace	
*main filter				X				replace	
9. Blower ground brush						X		replace	
10. Spindle ground brush						X		replace	
11. Blower drive belt								replace	P/ECEM Manual

COMPUCHEM LABORATORIES

PROJECTED PREVENTIVE MAINTENANCE SCHE:

FINNIGAN GAS CHROMATOGRAPH / MASS SPECTROMETER

JANUARY 1990 TO JUNE 1990

UNIT #	NORMAL ROUTINE	DUE FROM PREVIOUS ***	JAN	FEB	MAR	APR	MAY	JUN
01	04-08-12					DUE		
02	03-07-11				DUE			
03	01-05-09		DUE				DUE	
04	03-07-11				DUE			
05	03-07-11				DUE			
06	01-05-09		DUE				DUE	
07	02-06-10			DUE				DUE
08	02-06-10			DUE				DUE
09	04-08-12					DUE		
10	04-08-12					DUE		
11	02-06-10			DUE				DUE
12	02-06-10			DUE				DUE
13	01-05-09		DUE				DUE	
14	01-05-09		DUE				DUE	
15	04-08-12					DUE		
16	01-05-09		DUE				DUE	
17								
18	03-07-11				DUE			
19	03-07-11				DUE			
20	03-07-11				DUE			
21	02-06-10			DUE				DUE
22	04-08-12					DUE		
23	02-06-10			DUE				DUE

6.5 Material Procurement and Control

CompuChem's Purchasing Department has two prime objectives: (1) maintain sufficient supplies of all required items as needed, and (2) encourage all forms of competition in order to aggressively seek the best total value in a combination of supply, price, required quality, and service.

Procedures for Purchasing Materials: Department and laboratory managers have primary responsibility for maintaining adequate inventory of supplies and ensuring that all supplies/equipment meet or exceed quality requirements. Managers work through the Purchasing Department to accomplish these objectives. CompuChem® uses competitive inquiries or requests for bids, along with appropriate negotiation, to provide equal opportunities for potential and current suppliers to earn CompuChem's business and to allow the laboratory to seek the best total value. Long-term considerations include reliability, price, required quality and service. Vendors are encouraged to bring to CompuChem's attention new or improved materials, equipment and services. Suppliers must maintain the confidentiality of competitively sensitive information which is obtained from the Purchasing Department or other CompuChem® personnel. Prices and related information, whether accepted or not, will not be disclosed.

Each year, various vendors will supply the laboratory with solvent/chemical samples during the bidding process. The laboratory evaluates each vendor's sample, as described in the next section, before the bid is considered by the Purchasing Department. If solvent/chemical quality is equivalent, then price and service are considered. Prices are kept low because of the highly competitive market and the high volume used by the laboratory.

Material Quality Inspection: Managers interact with the Quality Assurance Department when purchasing supplies/equipment that could potentially affect data quality, and therefore testing prior to use in the laboratory. The Director of QA (or designated QA staff member) determines the appropriate test procedures and evaluates the resulting test data. A similar validation process is used in testing new instrumentation, as described in Appendix A. All new lots of standards, reagents, and glassware are tested as described in section 9.2 of the QA Plan and in the QA SOP Manual. The quality testing of solvents and other high-volume chemicals is described further in the following section. Additionally, the laboratory continually evaluates the integrity of these materials by performing the routine QC method blanks with every sample batch as described in sections 9.2 and 9.3 of the QA Plan.

When variability is exhibited in the quality of vendor-supplied materials or services, the laboratory/department manager is responsible for working with the Purchasing Department to find a suitable alternative.

Chemical and Standard Inventory Procedures: All chemicals other than organic standards are inventoried by appropriate laboratory manager and re-ordered as needed, with adequate time allowed for order processing, shipment and quality testing. The vendor supplying extraction solvents first provides a test sample from a particular solvent lot. After testing by the laboratory, if the lot is approved, several cases of the lot are purchased, with the remaining cases of the same lot stored by the vendor. The vendor is responsible for keeping inventory of the solvent lot, and when only a few cases remain, provides the laboratory with a test sample from a new lot. The process is repeated so that

a second approval lot is immediately available once the first is consumed. The lab manager maintains files of all test data to verify solvent purity. All extractions are traceable to the approved solvent lot used in sample preparation.

Organic standards are prepared internally in the Standards Laboratory, and the Standards Laboratory Chemist is responsible for maintaining adequate inventories and initiating standard purity testing and tests of each standard preparation lot, as described in section 9.2 of the QA Plan and in the QA SOPs.

Solvent Storage and Waste Disposal: All solvents in use in the laboratory are kept in solvent storage cabinets, which are vented and specifically designed for this use. Acids are kept in a separate, specially designed storage cabinet. Solvents and other chemicals not in use are stored in the Solvent Storage Room, described in section 6.2. Periodically, a waste disposal firm removes the laboratory's waste, stored in 55-gallon drums, to a licensed hazardous waste landfill.

liquid waste to a landfill ?

6.6 Glassware Preparation/Decontamination

Laboratory Glassware: Glassware used in the Organic Sample Preparation Laboratory is washed initially by hand in a hot, nonphosphate, laboratory-grade detergent solution, then loaded onto racks and into a stainless steel industrial dishwasher. When the normal wash cycle is completed, the rinse cycle is started and the glassware is rinsed continuously (approximately 5 minutes) with ordinary tap water. This is followed by a second rinse cycle (approximately 5 minutes) using laboratory-pure (distilled, deionized) water. The rack is then removed from the dishwasher, loaded onto aluminum trays and annealed at 500°C for six hours. Glassware used for Inorganic Sample Preparation and volumetric glassware, such as pipets and flasks, are not annealed but instead air-dried while inverted. After annealing or air-drying, the decontaminated glassware is loaded onto large carts which are labeled and segregated from unprepared glassware. Inorganics glassware is loaded onto a clean Inorganics Station (cart) and distributed directly to the lab.

SampleSaver® Glassware: SampleSaver® glassware is prepared separate from all other laboratory glassware, and the procedures differ slightly, depending on the type of container. One-liter glass bottles, depending on their condition, may be recycled. In such cases, they are washed, rinsed and dried as described above, but separate from all other glassware. After this step, they may be mixed with unused glassware and the procedure repeated as described above for laboratory glassware. The glassware is baked at 230-260°C for one hour, then loaded onto clean carts for cooling. Once cool, they are capped, loaded into boxes (those used for shipping the unused containers from the vendor to the lab) and stored for later use.

For 500 ml plastic containers used for metals and Mercury sample collection, the containers are soaked for one hour in a 50% nitric acid solution following the soap solution wash. After draining, the containers are rinsed three times with tap water, then three times with laboratory-pure water. The bottles are inverted and air-dried, capped, then stored in the Inorganics SampleSaver® cabinet in the Glassware Preparation area.

Glass bottles used for volatiles, Cyanides and Phenols sample collection (40 ml) are prepared in the same manner as the liter containers described above, but are baked at 260°C for one hour. The bottles are removed from the oven as soon as they are cool enough to touch, then capped immediately with septum-sealed, Teflon-lined caps and stored in a contaminant-free cabinet.

Plastic caps, Teflon discs (cap liners) and Teflon-lined septa are washed and rinsed in the same manner as the glassware above, but are baked for one hour at 105°C. Following a brief cooling period (cool enough to touch), the discs and septa are placed into their caps and used to seal the cleaned liter and 40 ml bottles.

APPENDIX B

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR CHROMIUM VI ANALYSIS AND OTHER INFORMATION

APPENDIX B

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURE FOR CHROMIUM VI ANALYSIS AND OTHER INFORMATION

TABLE OF CONTENTS

B.1	Additional Information on the Hexavalent Chromium Standard Operating Procedure
B.2	Hexavalent Chromium Standard Operating Procedure
B.3	Chain-of-Custody Procedures
B.4	Standard Operating Procedures for Data Package Assembly
B.5	Data Reduction, Validation, and Reporting
B.6	Performance and System Audits

APPENDIX B.1

EMS HERITAGE LABORATORIES, INC.

ADDITIONAL INFORMATION ON THE HEXAVALENT CHROMIUM STANDARD OPERATING PROCEDURE

ADDITIONAL INFORMATION ON THE HEXAVALENT CHROMIUM SOP

The following additional information applies to the attached SOP for Hexavalent Chromium analysis.

1. Sample matrix: water.

2. Sample size: 250 ml.

3. Preventative maintenance: the spectrophotometer is checked quarterly by checking the wavelength with a standard filter.

4. Calibration standards: the calibration standards are prepared from Fisher (or equivalent) $K_2Cr_2O_7$ crystals at the following concentrations: 0.005, 0.010, 0.025, 0.05, 0.1, 0.25, and 0.5 ppm. The standards are stored at room temperature for no more than 6 months.

5. Quality Control samples codes:
   - BLA01: Blank (distilled water containing same reagents as the sample.)
   - LCS02: Laboratory control sample (0.2 ppm, prepared from 1000 ppm Fisher stock solution.)
   - DUP01: Duplicate
   - SPI01: Spike (0.025 ppm, prepared from 1000 ppm Fisher stock solution.)
   - VER01: Continuing calibration standard (0.025 ppm, prepared from 1000 ppm Fisher stock solution.)

6. Data deliverables: the final data package will included the following items:

   Table of contents
   Case narrative
   Initial and continuing calibration results
   Raw data
   Calculated results
   QC sample results
   Chain-of-custody records

7. Chain-of-custody procedures: see attached SOPs.

APPENDIX B.2

ERM HERITAGE LABORATORIES, INC.

HEXAVALENT CHROMIUM STANDARD OPERATING PROCEDURE

EMS Test Code: M310.6
Referenced Methods:
APHA 312 A
SW846 7196
Rev. #1

HEXAVALENT CHROMIUM

I. INTRODUCTION

Chromium may exist in two forms, trivalent and hexavalent. The hexavalent form is more commonly occurring, and it is also the form of major concern. Hexavalent Chromium has a corrosive effect on skin and may produce ulcers on prolonged contact. Hexavalent chromium is a known carcinogen. Chromium is used predominantly in industrial processes and may enter water supplies through waste discharge. Compounds containing chromium are also added to cooling water lines to help prevent corrosion.

A.	Detection Limit	0.01 mg/l
B.	Regulatory Limit	Discharge to waterways 0.1 mg/l (Illinois) General standard for the waters of Illinois 0.05 mg/l Pretreatment limit will vary according to industrial user category and POTW requirements
C.	Holding Time	24 hours
D.	Preservative	Cool, 4° C, not filtered
E.	Special Considerations	Hexavalent Chromium has a holding time of only 24 hours so samples must be analyzed as soon as possible after sampling, as hexavalent chromium is reduced to the trivalent form over time.

II. APPARATUS AND REAGENTS

A. Spectrophotometer, 540nm

B. Plastic cups

C. 25 ml graduate cylinder

D. 1+1 H_2SO_4

Preparation: Using a volume/volume dilution, dilute one part sulfuric acid to one part water. CAUTION: Always add acid to water, never the reverse. This reagent should be prepared in a cold water bath, as intense heat is generated.

E. Powder pillows chrome Ver# (HACH Chemical Co.)

F. Stock Hexavalent Chromium - 50 mg/l (1 ml = 0.05 mg)

Preparation: Analytically weigh 141.4 mg $K_2Cr_2O_7$, dissolve and bring up to volume in a one liter volumetric flask. The concentration of this solution is 50 mg/l Hexavalent Chromium. Holding time 6 months. Use caution when handling this reagent it is a known carcinogen.

III. PROCEDURE

A. Theory

Hexavalent Chromium in this procedure is determined colorimetrically by reaction with diphenylcarbazide in an acidic solution. A redviolet dye is produced which can be measured spectrophotometrically at 540 nm.

B. Method

1. Turn on the spectrophotometer and set wavelength to 540 nm, and allow instrument to warm up for 15-20 minutes.

2. Use 25ml of sample and standards.

3. Add 2 - 3 drops 1+1 H_2SO_4 and swirl.

4. Add 1 powder pillow and swirl until powder has dissolved

(approximately 30 seconds).

5. Read absorbance.

IV. QUALITY CONTROL

A. A curve of at least 5 standards will be generated. Linear range is to 1 ppm.

B. BLA01 - once per run. Must be below detection limit reported.

C. LCS02 - 1 per run, recovery must be 100% ± 10%.

D. DUP01 - 1 per run or 10 samples. RPD must be less than 20% or 5x D.L. ± D.L.

E. SPI01 - 1 per run or 20 samples, recovery must be 100% ± 20%.

F. VER01 - Every 10 samples, mid range recovery must be 100% ± 10%.

V. CALCULATIONS

Samples concentration is calculated from a linear regression curve of the standards. Multiply this value times any dilutions to get final sample concentration.

mg/l (off curve) x Dilution = mg/l Hexavalent Chromium

VI. TROUBLESHOOTING/INTERFERENCES

A. If the sample has any appreciable color or turbidity of it's own, it will rad a falsely high absorbance. This can be corrected by running a "color blank", which is the sample's absorbance without any reagents added. This value is subtracted from the sample absorbance to yield a corrected value.

Sample absorbance - Color Blank absorbance = Corrected Sample absorbance.

B. The diphenylcarbazide reaction is nearly specific for chromium, therefore chemical interferences are uncommon.

Hexavalent molybdenum and mercury will react to form a color in solution with diphenylcarbazide but the lower pH of the chromium analysis will keep the color intensity low and interference is unlikely. Iron concentrations greater than 1 mg/l will produce a yellow color but again the color is generally

not intense and therefore usually provides no difficulty in analysis. Vanadium can interfere strongly but concentrations up to 10 times that of chromium can be tolerated.

VII. SAFETY

Hexavalent Chromium is a known carcinogen.

1 + 1 H_2SO_4 is corrosive

Safe laboratory practices should be used at all times

VIII. CLEAN UP

The Hexavalent Chromium samples and standards may be disposed of down the sink when flushed thoroughly with water.

9002\jk10m

APPENDIX B.3

EMS HERITAGE LABORATORIES, INC.

CHAIN-OF-CUSTODY PROCEDURES

RECEIVED DEC - 6

2.0 THE DUTIES AND RESPONSIBILITIES OF SAMPLE CUSTODIANS

Duties and responsibilities of the sample custodian shall include but not be limited to:

2.1 Receiving samples

2.2 Inspecting sample shipping containers for presence/absence and condition of :

2.2.1 custody seals, locks, "evidence tape," etc.
2.2.2 container breakage and /or container integrity

2.3 Recording condition of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or fields in the computer lab information management system (LIMS), and/or on appropriate client forms.

2.4 Signing appropriate documents shipped with samples (i.e. airbills, chain-of-custody record(s), etc.)

2.5 Verifying and recording agreement or non-agreement of information on sample documents (i.e., sample tags, chain-of-custody records, traffic reports, airbill, etc.) in appropriate logbooks or computer fields and/or on appropriate forms. If there is non-agreement in these documents, recording the problems, contacting the client for direction and informing the appropriate laboratory personnel. (The corrective action directions shall be documented in the project file.)

2.6 Initiating the paper work for sample analyses on appropriate laboratory documents and/or in the laboratory data system as required for initiation of the analysis sequence according to standard laboratory procedures.

2.7 Marking or labeling samples with laboratory sample numbers generated by the computer and cross referencing laboratory numbers to client numbers using the appropriate fields in the computer log-in procedure and/or chain-of-custody sheet.

2.8 Placing samples and spent samples into appropriate storage and/or secure area.

2.9 Controlling access to samples in storage and assuring that laboratory standard operating procedures are followed when samples are removed from and returned to storage.

2.10 Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination. The temperature of the sample storage areas recorded daily and any deviations from 4 ± 2 degrees C noted. Samples are opened (when necessary) under a fume hood individually to avoid cross contamination.

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Preservation of volatile vials with HCl will be done under the charcoal fume hood in the mass spec VOA prep lab (lab #11) and the date, lot number and other information entered into the logbook.

2.11 Returning shipping containers to the proper sampling teams.

2.12 Maintaining custody of the sample tracking logbook.

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3.0 STANDARD OPERATING PROCEDURES FOR SAMPLE RECEIVING

3.1 Upon receipt, examine the shipping container and record the following information in the computer LIMS system or on an appropriate form.

3.1.1 Presence/absence of custody seal(s) on the shipping containers(s).

3.1.2 Custody seal number(s) and condition (i.e. intact, broken, absent) when present.

3.2 Open the shipping container, remove the enclosed sample documents and record the presence/absence of the following forms and their respective numbers in the LIMS system or on an appropriate form:

3.2.1 The chain-of-custody record(s).

3.2.2 Record the project identification numbers.

3.3 Remove sample containers and record in the LIMS system or the appropriate form:

3.3.1 Condition of samples (intact, broken, leaking, etc).

3.3.2 Presence/absence of sample tags.

3.3.2.1 Record sample tag document control numbers.
3.3.2.2 Compare with chain-of-custody record(s).
3.3.2.3 Document whether or not these numbers agree.

3.4 Compare the documents listed below to verify the information contained on them. Both agreement among the forms and any discrepancies found will be documented. If discrepancies are found, contact client or project manager for clarification (and/or resolution) and notify appropriate laboratory personnel. The documentation of corrective action instructions and the source of those instructions will be forwarded to the Project Manager for inclusion in the data file.

3.4.1 Chain-of-custody records

3.4.2 Sample tags

3.4.3 Laboratory Task Order

3.5 If all samples recorded on the chain-of custody record were received by the lab and there are no problems observed with the sample shipment, the custodian will sign the chain-of-custody record in the "received for laboratory by:" box.

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BY Kym DATE 12/5/91

If problems are noted, sign the chain-of-custody record and then note problems in remarks box or reference other forms that describe the problems in detail.

3.6 The sample custodian should record the laboratory sample numbers assigned by the computer on the sample containers and tags.

3.7 Samples received when the sample custodian and other designated recipients are absent will be placed in the proper lab location. The person receiving the shipping container will sign for the container, place it in the secure, refrigerated location and record the time, date and name of individual receiving the container.

The sample custodian or their designee will log-in the samples on the next business day. The date on the receipt form will be the date the form was completed.

3.8 Broken samples should be disposed of appropriately and the client notified that re-sampling is necessary.

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4.0 STANDARD OPERATING PROCEDURES FOR SAMPLE IDENTIFICATION

4.1 In order to maintain sample identity each sample received must have a unique sample identification number. The laboratory identification number is a sequential number assigned by the LIMS computer. The LIMS system allows for the cross-reference of laboratory sample number and client number along with date of sample receipt.

4.2 Sample containers (bottles and extract vials) will be clearly identified with the appropriate sample number.

4.2.1 During the sample receiving process, the sample custodian or other appropriate personnel will assure that each sample container is identified with a unique sample ID number and that number is recorded in the sample receipt documentation. The laboratory ID number, assigned by the LIMS system, is written on the sample container label and cap.

4.2.2 Extract vials will have the sample ID numbers and the appropriate fraction (Acid, Base, Pest etc.) written on the vial. These numbers should be recorded on the sample tracking documentation (benchsheets).

5.0 STANDARD OPERATING PROCEDURES FOR SAMPLE STORAGE

5.1 The samples will be removed from the shipping container and stored in their original containers unless damaged.

5.2 Damaged samples will be transferred to another bottle or disposed of in an appropriate manner and this transferral/disposal will be documented in a logbook along with the date and initials of the analyst.

5.3 Samples and extracts will be stored in a secure area, on the shelves designated for that analysis or project.

5.4 The secure areas include the walk-in refrigerator #16 for Semi-volatiles and PCB/Pest, and refrigerator #25 general inorganic and metal samples and the refrigerators in the Mass Spec (VOA samples) and GC (Pest extracts) rooms. These areas are locked after normal working hours. These storage areas are maintained at 4 ± 2 degrees C. The temperature is monitored and recorded daily, by the receiving personnel or group.

5.5 The storage area will be kept secure at all times. The sample custodian will control access to the storage area through the receiving area and the building security system controls access from other doors.

5.6 Whenever samples are removed from storage, this removal will be documented in the proper logbook (extraction, analysis). The date of extraction or analysis and the analyst's initials will be entered into the LIMS system to permit sample tracking throughout the analysis process.

5.7 Samples and extracts will be stored after completion of analysis in accordance with the contract or until instructed otherwise by the client. At this time, samples are stored for 30 days after reporting and extracts for 90 days.

5.8 Standards will not be stored with samples. The working standards for PCB's/Pests, Semi-volatiles and Volatiles are stored in refrigerators in each analytical lab.

5.9 In order to satisfy sample chain-of-custody and confidentiality requirements, the following standard operating procedures will be followed.

5.9.1 Samples will be stored in a secure area.

5.9.2 Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked and access limited to employees only.

5.9.3 Visitors will sign a visitors log and be escorted by a plaza employee while in the laboratory. No laboratory area will be entered unless a member of the laboratory staff is present.

5.9.4 Refrigerators, freezers and other sample storage areas will be securely maintained or locked.

5.9.5 Only the designated sample custodian and the supervisory personnel of each lab group will have keys to locked sample storage area(s).

5.9.6 Samples will remain in secure sample storage until removed for sample preparation or analysis.

5.9.7 All transfers of samples into and out of storage will be documented in the appropriate analysis logbook.

5.9.8 After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the sample/extract to the storage area before the end of the working day or prior to the end of his/her shift.

6.0 STANDARD OPERATING PROCEDURES FOR TRACKING SAMPLE ANALYSES

6.1 Both the preparation and the analysis of samples will be documented.

6.2 All analysts will use the proper bench sheet for the analysis.

6.3 All notebook pages, bench sheets, graphs, computer printouts, chromatograms, GC/MS spectra and other laboratory project related documents will contain the date, signature (initials) of the analyst and other pertinent information as stated in the method or contract.

6.4 Upon completion of analysis, data will be filed in the appropriate sample files.

6.5 Copies of QA/QC will be placed in the appropriate file.

6.6 Instrument logs will be maintained for each gas chromatograph-mass spectrometer (GC/MS), gas chromatograph (GC), inductively coupled plasma (ICP) unit, atomic absorption unit (AA), and wet chemistry instrumentation equipment (e.g. CN-distillation apparatus, UV/VIS, colorimeter, etc). This logbook will contain the order of analysis, the instrument parameters, maintenance performed (and why) etc.

6.7 Copies of the instrument logs will be placed in the appropriate files, as necessary.

6.8 All sample preparation information will be documented in the laboratory notebook or bench sheet. All sample analysis data will be documented using instrument logbooks, computer printouts, chromatograms, analyst's laboratory notebooks, and/or other laboratory documents. When sample preparation or analysis is finished, the completed documents should be initialed and placed in the appropriate sample and/or project files.

6.9 If samples are run in batches, which may include several projects, all original batch analysis results will be filed in one sample/project folder. Copies of the results will be placed in each of the other sample/project folders and there shall be a reference to the file that contains the original analytical results documentation. Original calibration and QA/QC data should be treated in the same manner as the analytical documentation.

6.10 If, for any reason the original data cannot be placed in a sample/project file, this shall be documented and the location of the original data noted on the file inventory (xerox copies of the data will be included in the file).

6.11 All notes, comments and calculations made on or added to, project related data/documents will be signed and dated by the author/analyst/reviewer.

APPENDIX B.4

EMS HERITAGE LABORATORIES, INC.

STANDARD OPERATING PROCEDURES FOR DATA PACKAGE ASSEMBLY

7.0 STANDARD OPERATING PROCEDURES FOR DATA PACKAGE ASSEMBLY

7.1 A procedure for organization and assembly of all documents relating to each client will be implemented by the appropriate Project Manager or designated person.

7.2 This procedure will ensure that all documents are compiled in one location for submission, and are arranged in the correct order.

Prepare data file folders:

7.2.1 Using appropriate file folders, assign one folder to each project.

7.2.2 Place all documents pertaining to one project in the folder.

7.2.3 Documents should be arranged by document type within the folders, i.e., all ICP data, all hard-copy Mass spectra together and in order as described in the contract or method.

7.2.4 These document files will be filed in one location and stored in a secure area.

7.2.5 The kinds of documents that will be contained in project and sample files are described in Tables 1 and 2.

7.3 The system must include a document numbering and inventory procedure.

7.3.1 Each document type (see TABLE 1 and 2) is inventoried and listed on the cover sheet of the report. The number of pages and any unique identifier is listed.

7.3.2 All documents pertaining to each project including, but not limited to, the following will be numbered and inventoried. All document groups should be clipped together. A document inventory list can be used for extensive projects (TABLE 3).

7.3.2.1 Custody (internal and chain-of-) records, weekly reports.

7.3.2.2 Laboratory logbooks, personal logbooks, instrument logbooks,bench sheets.

7.3.2.4 Laboratory data (sorted by sample, when possible), calibration and quality control results.

7.3.2.5 Data summaries and reports.

DO NOT
DUPLICATE

7.3.2.6 All other documents, forms, or records referencing the samples.

7.3.3 Preparation of a document inventory

7.3.3.1 A document inventory list provides a record of all documents, and their corresponding document numbers, that are included in the completed file.

7.3.3.2 A separate document inventory list (See Table 3) is prepared for each project.

7.3.3.3 The laboratory will retain copies of the document inventory lists for files reported to the client.

DO NOT
DUPLICATE

TABLE 1

ORGANICS DOCUMENTS

File inventory

* Chain-of-custody

Organics Analysis Data Summaries

Bench sheets and worksheets

Copies of instrument logbook pages

Sample tracing documents

Sample receipt logbook pages

** Internal custody records

Hard copies of mass spectra and chromatograms

QA/QC package

DFTPP/BFB calibration spectra and worksheets

Quality control reports

Standards analysis report forms, worksheets, spectra and chromatograms

Duplicate, matrix, surrogate spike results

GC/MS computer library search worksheets and accompanying spectra

Related correspondence and/or memos

All other related documents

---

* If received with sample shipment
** If used to supplement sample tracking system

Each file must contain these documents or a memo explaining their absence (one memo may cover several documents).

TABLE 2

INORGANICS DOCUMENTS

File inventory

* Chain-of-custody

Inorganics Analysis Data Summaries

Copies of analysts' notebook pages and/or bench sheets

Worksheets

ICAP and AA instrument logbook pages

Sample tracing documents

Sample receipt log pages

** Internal custody records

Copies of instrument printouts

QA/QC data reports

Duplicate, blank, etc. analyses result

Related correspondence and memos

All other related documents

---

* If received with sample shipment.
** If used to supplement sample tracking system.

Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents).

TABLE 3

DOCUMENT INVENTORY (EXAMPLE)
Client No. 0000

Document Control #	Document Type	# Pages
Client#-(Project)#-Serial#		
0000-X-0001	Project File Document Inventory Sheet	__
0000-X-0002	Case (Project) Narrative	__
0000-X-0003	*Chain-of-custody	__
0000-X-0004	Benchsheets	__
0000-X-0005	Instrument Logbook Pages	__
0000-X-0006	Sample Receipt Logbook Page	__
0000-X-0007	Internal Custody Records	__
0000-X-0008	ICP Data for Sample ######	__
0000-X-0009	Volatile Data for Sample ######	__
0000-X-0010	Semi-volatile Data for Sample ######	__
0000-X-0011	GC Chromatograms for Sample ######	__
0000-X-0012	Cyanide Run Sheet for Samples ######-######	__
0000-X-0013	Calibration Information (for each instrument)	__
0000-X-0014	Quality Control Reports (Blanks)	__
0000-X-0015	Standards Spectra and Chromatograms	__
0000-X-0016	Matrix Spike/Spike Duplicate data	__
0000-X-0017	All other related documents	__

* If received with sample shipment
Note: Each file must contain the following documents or a memo explaining their absence (one memo may cover several documents).

0000-X-0001

APPENDIX B.5

EMS HERITAGE LABORATORIES

DATA REDUCTION, VALIDATION, AND REPORTING

DEC 9
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12.0 Data Reduction, Validation and Reporting

12.1 Introduction

The laboratory analyst holds the key to the successful completion of valid and documentable analytical testing. Analysts must be provided with proper training, equipment and supervision so that they know and follow proper analytical procedures. It is management's responsibility to make available at the bench level, this QA Plan and it is the analysts' responsibility to know and to follow its' requirements. In addition to the QA Plan, pertinent EMS Heritage SOPs, EMS Heritage written methods and published (EPA, etc.) methods, the analysts must be fully aware and have easy access to any specific contract requirements and DQO's. Data validation will never result in acceptable data if the proper QC types are not analyzed as dictated by the specific project or contract.

12.2 Data Reduction

This section outlines how the raw data are reduced (calculated) into reportable values.

12.2.1 All lab analysts share some portion of the responsibility for maintaining documentation of the testing being performed. In addition to the various bound notebooks which are maintained documenting standard preparations, instrument maintenance, instrument performance, logbook of logbooks, etc., analysts must maintain some combination of bound notebooks and/or original raw data printouts with bench sheets indicating the analytical run contents, sequence and results. Some documentation requirements under various testing programs may make the use of loose bench sheets in lieu of bound sequentially numbered notebooks an unacceptable procedure, in which case bound notebooks will be utilized exclusively. Exclusive use of bound notebooks may necessitate the use of loose sheets for data entry of results and associated information into the LIMS* system.

12.2.2 All analytical run notebooks must contain the following information at a minimum:

1. Analyst identification

2. Date of analysis

* Laboratory Information Management System

3. Instrument identification

4. Analytical method (Edition or Version) used and any deviations

5. Date of initial calibration

6. Computer file name if applicable

7. Concentration and source identification of all standards and spikes

8. EMS sample I.D. number

9. Reference to prep performed

10. Any comments pertinent to the analysis or sample conditions (homogeneity)

11. Any dilutions, concentrations, sample manipulations (initial and final weights/volumes, etc.)

Calculation of results from raw data if not contained in the bound lab notebook, will be maintained and filed along with the original hard copy of instrument printouts.

12.2.3 When data entry of results into LIMS is required (direct data file transfer not available), it will be necessary to summarize the final results and all information needed for data entry. A combination of the analytical run notebook, the instrument printouts/chromatograms, calculation work sheets, prep bench sheets/notebooks and summary of final results will be sufficient to re-construct the sample analysis. This simple final result summary will include at a minimum:

1. Analyst identification

2. Date of analysis

3. Instrument identification

4. Analytical method used

5. Computer file names

6. EMS sample I.D. number

7. Comment to appear on the final report

8. Any dilutions or concentrations involved

9. Final results with proper units and detection limits

10. Reviewer initials and date of review

11. Data entry operator and date of data entry

12.2.4 All instrument printouts/chromatograms must contain the following at a minimum:

1. Analysts' initials and date of review of hard copy

2. Sample or standard identification (and conc.)

3. Computer file numbers

4. Dilutions or concentrations

5. Date and time of injection or analysis

6. Units (µg/L, mg/L, mg/kg, etc.)

7. Identification of samples that are spiked or duplicated (in addition to computer assigned "Q" numbers).

8. Peaks chosen for quantitation and their identification.

12.2.5 Some testing will generate only bench sheets when no instrument printout is possible or practical (pH, titrations, etc.) and no bound notebook is maintained. In those cases this bench sheet must contain all information required in the analytical run notebook previously discussed <u>and</u> the following at a minimum:

1. Reviewer initials and date of review

2. Data entry operator initials and date of entry

3. Page numbers

4. Calculated final results, %R of spikes, RPD of duplicates, etc.

5. Any raw data used to arrive at the final result

6. Results of the calibration curve (linear regression, log transforms, etc.)

12.2.6 All calibration curves conforming to a linear equation will be fitted by the least squares linear regression technique. This procedure will also be used for calibration curves that require log transforms or similar transforms. The formula for calculation of the regression line is as follows:

Where: $Y = mX + b$
Y = Point on the y axis
m = Slope of line
X = Point on the x axis
b = y intercept

The least squares estimate of b and m uses the following equations:

$$m = \frac{\sum XY - \frac{\sum X \sum Y}{n}}{\sum X^2 - \frac{(\sum X)^2}{n}}$$

$$b = \bar{Y} - m\bar{X}$$

Other scientifically sound procedures/data reduction techniques may be used as permitted by the Chief Chemist and the QA Officer.

12.2.7 When using the external standard calibration technique the average calibration factor (CF) can be used in place of a calibration curve (non-linear) when the %RSD is less than 20% over the working range and linearity through the origin can be assumed. The calibration factor (CF) is calculated as follows:

$$CF = \frac{\textit{Total Area of Peak*}}{\textit{Mass Injected (in nanograms)}}$$

or $$RF(\textit{Response Factor}) = \frac{\textit{Mass injected (in nanograms)}}{\textit{Total Area of Peak*}}$$

*For multi-response pesticides/PCBs, gasoline, diesel, etc., use the total area of all peaks used for quantitation.

The concentration of each analyte in the sample may be determined by calculating the amount of standard purged or injected, from the peak response, using the calibration curve or the calibration factor (CF) above. The concentration of a specific analyte is calculated as follows:

<u>Aqueous samples</u>:

$$\text{Concentration (ug/L)} = [(A_x)(A)(V_t)(D)]/[(A_s)(V_i)(V_s)]$$

where:

A_x = Response for the analyte in the sample, units may be in the area counts or peak height.

A = Amount of standard injected or purged, ng.

A_s = Response for the external standard, units same as for A_x.

V_i = Volume of extract injected, μL. For purge-and-trap analysis, V_i is not applicable and therefore = 1.

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

V_t = Volume of total extract, μL. For purge-and-trap analysis, V_t is not applicable and therefore = 1.

V_s = Volume of sample extracted or purged, mL.

By inspection of the above formula one observes that the concentration in the extract is:

$$(A_x)(RF)$$

where: $$RF(Response\,Factor)^* = \frac{A}{A_s}$$

*RF is the inverse of CF.

Nonaqueous samples:

$$\text{Concentration (ng/g)} = [(A_x)(A)(V_t)(D)]/[(A_s)(V_i)(W)]$$

where:

W = Weight of sample extracted or purged, g. The wet weight or dry weight may be used, depending upon the specific applications of the data.

A_x, A_s, A, V_t, D, and V_i have the same definition as for aqueous samples.

12.2.8 When using the internal standard technique (all GC/MS analyses) the average response factor ($\overline{RF}$) can be used in place of a calibration curve (non-linear) when the %RSD is less than 20% over the working range and linearity through the origin can be assumed. The response factor (RF)* is calculated as follows:

$$RF = (A_s C_{is})/(A_{is} C_s)$$

where:

A_s = Response for the analyte to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard, μg/L.

C_s = Concentration of the analyte to be measured, μg/L.

*Also known as "Relative Response Factor" or RRF

The concentration of a specific analyte is calculated as follows:

Aqueous samples:

$$\text{Concentration } (\mu g/L) = [(A_x)(C_{is})(D)]/[(A_{is})(RF)(V_s)]$$

where:

A_x = Response of the analyte being measured, units may be in area counts or peak height.

C_{is} = Amount of internal standard added to extract or volume purged, ng.

D = Dilution factor, if a dilution was made on the sample prior to analysis. If no dilution was made, D = 1, dimensionless.

A_{is} = Response of the internal standard, units same as A_x.

RF = Response factor for analyte.

V_s = Volume of water extracted or purged, mL.

Nonaqueous samples:

$$\text{Concentration } (\mu g/kg) = [(A_x)(C_{is})(D)]/[(A_{is})(RF)(W_s)]$$

where:

W_s = Weight of sample extracted, g. Either a dry weight or wet weight may be used, depending upon the specific application of the data.

A_x, C_{is}, D, A_{is}, and RF have the same definition as for aqueous samples.

The RF from the daily standard (CCV) analysis is used to calculate the concentration in the sample unless specific methods dictate otherwise.

12.2.9 Manual data entry is the responsibility of the Data Entry Supervisor who will assure that data entry staff have knowledge of the system in order that complete analytical information is entered accurately, in the correct format and labeled consistently. Data entry staff will not perform any calculations; all data entry items will be summarized and submitted on standard forms which have been reviewed by the responsible group leader for acceptability, accuracy and completeness.

All analyses in the data system are based on the "analytical run" concept. All data (prep or analytical, is grouped in the computer, exactly in the order analyzed (as it also appears on a benchsheet). All QC is included in the analytical sequence.

Automated data transfer is the responsibility of each respective Group Leader.

12.3 Data Validation - During Collection

The principal criteria to be used to validate data integrity during collection will be the following:

1. Calibration Criteria (Linearity, RF etc.)
2. Internal Standard Areas (organics)
3. Calibration Blank Value (CAL01)
4. Method Prep Blank Value (BLA02)
5. Reagent Blank Value (BLA01)
6. Result of calibration verification standard used to verify calibration (ICV01).
7. Results of (matrix) spiked non QC samples (DPS01, SPI01, SPI02, DPS02).
8. Results of surrogate recoveries (organics)
9. Results of replicate measurements (DUP01, DUP02, DPS02).
10. Results of Continuing Calibration Verification standard (CCV)

11. Results of Laboratory Fortified Blanks and Laboratory Control Samples or QC Check Samples (LCS, DLCS, ICV02)

12.3.1 These measurements are to be made by the analyst who, using specific acceptance criteria, will either proceed with analyses or take corrective action. All QC data must be reviewed by the Group Leader to insure that all QC types have been completed and meet acceptance criteria. Group Leaders must also:

1. Check raw data entries and calculations.

2. Check extraction logs and benchsheets.

3. Check Calibration integrity.

4. Check Instrument/analytical logbooks.

The Group Leaders are responsible for assuring that all bench sheets submitted for data entry are accurate and adequate to allow for the rapid and error free recording of data into LIMS.

12.3.2 Group Leaders and the Sample Custodian are also responsible for checking and assuring that all internal chain of custody requirements and documentation of such are met.

12.3.3 In summary, Group Leaders will review all bench sheets or release computer files to LIMS after review. In addition 20% of all raw data will be reviewed by the Group Leaders to insure that the method was in-control during the analytical run. Group Leaders are responsible for initialing and submitting data for data entry upon completion of their review. This completes the first step in review of the data integrity and data validity.

12.4 Data Validation - Post Completion

Subsequent to data entry, the Quality Assurance Unit is responsible for comparing bench sheets with actual data entered into LIMS. Such review will occur for 2-5% of all data entered. This activity will be documented and retained on file.

Refer to Figure 12.1, Data Review Protocol for the procedures used at EMS Heritage for data review and validation. The QA Officer and/or the QA Unit must also:

1. Confirm that the goals for precision and accuracy on duplicate and spiked compounds are sufficient to achieve the goal of 99% completeness.

2. Confirm that all analytical QC precision and accuracy (the measurement system, not sample (matrix) related) is 100% acceptable or that the run has been rejected and corrective action taken.

3. Confirm that all client specific or contract specific QC requirements have been met.

4. Examine at least 5% of the raw data (e.g. chromatograms, AAS recorder outputs, burn record on a periodic basis to verify adequacy of documentation, confirm peak shape and resolution, and to assure that the instrument system is sufficiently sensitive and responsive.

12.4.1 Internal and External Assessments

External assessment of the adherence to the QA/QC policy and procedures is the responsibility of the Quality Assurance Officer. Audit forms have been developed in the QAP and are used at all EMS Heritage Laboratories to evaluate their adherence to QA/QC Procedures. External assessments are also accomplished via third party audits.

Internal assessment of the compliance to company QA/QC policies and procedures is conducted by the Laboratory Director and the Quality Assurance Unit or their designee. Review of documentation, use of blind duplicates and standards and monthly reference materials shall be used to evaluate the performance of each laboratory. Refer to Section 14.0, Performance and Systems Audits for the details of internal and external assessments.

12.4.2 Quality Control Policy and Procedures

The EMS Heritage Laboratories, Inc. QAP is mandatory at all laboratories. Specific procedures are required and regular quality control audits are performed to assure compliance to company policy. Quarterly quality control reports are required from the QA Units submitted through the QA Officer. The overall quality of analysis shall be compiled and summarized by the QA Officer, who will report this summary to the President of EMS Heritage. Acceptable levels of

performance are presented and monitored by the QA Officer.

12.5 Laboratory Quality Control Checks

The types and frequency of laboratory QC Checks are presented in Table 11.5, Analytical Run Requirements/Frequency.* This table presents basically three categories of QC:

1. Instrument Calibration QC
2. Instrumental Method QC (tests w/no prep. or analyzed w/o prep)
3. Analytical samples QC - processed through all steps (prepped QC).

Instrument Calibration criteria must be met before any analyses are allowed to proceed.
Internal QC checks not related to calibration are discussed below.

12.5.1 Preparation or Method Blanks

Blanks are identified at EMS Heritage as either BLA01 or BLA02 (calibration blanks are treated as a calibration data point, CAL01). Those methods that do not differentiate a prep from the analysis, i.e. purge and trap volatile analyses, are labeled as BLA01, which is equivalent to a BLA02 for tests that do have a separable prep and analysis. The following control limits apply for all method blanks (BLA02) and reagent blanks (BLA01):

If sample results are greater than 10 times the method blank value or the sample results are below the detectable level, the analysis is acceptable. Otherwise, the following criteria will apply.

1. Organic Analytes: Less than the Reporting Detection level (PQL)

   Exceptions to the rule will be for common lab solvents not to exceed 5 times the PQL, including but not limited to the following:

   1. Acetone

* Only the page pertaining to Chromium VI analysis is included.

2. Methylene Chloride

3. Toluene

4. Methyl Ethyl Ketone (MEK)

5. Methyl Isobutyl Ketone (MIBK)

6. Freon(s)

7. Phthalates

2. Inorganic Analytes: Less than the Reporting Detection Level (PQL)

Exceptions to the rule will be for the following metals - not to exceed 3 times the PQL:

1. Iron

2. Nickel

3. Zinc

4. Copper

12.5.2 Any target compounds in the blank will be reported. Any sample containing a similar amount of the compound outside the acceptable range will be re-analyzed when the source of the contamination is identified and removed.

Target compounds found to be BDL (Below Detection Limit) in the blank will be entered into the data base at 0.4 times the MDL.

12.5.3 Matrix Spikes

Matrix spikes are included as a routine protocol according to the frequency given Table 11.5, Analytical Run Requirements/Frequency. Matrix spikes serve only to show that an individual sample does or does not exhibit matrix interferences using the prescribed method; they do not definitively demonstrate that a given analytical method is out of control. Other QC types will be utilized to demonstrate that an analytical method is in control (QC check sample, LCS, etc.).

12.5.3.1 There are basically two types of spikes utilized and they are as follows:

1. Pre-digestion/extraction spikes

2. Post digestion spikes

Pre-digestion/extraction spikes are performed for all applicable analyses at the required (Table 11.5) frequency. The goals for accuracy are listed in Table 5.1, QA Targets for Precision, Accuracy and Method Detection Limits.* Those control limits are determined from actual data entered into the QCTS when a sufficient number of observations (minimum of 7) are available to determine meaningful statistical limits.

Accuracy will be a function of the spiking level chosen. Many methods and testing programs specify the spiking levels to be used; EMS Heritage will use those spiking levels where specified. As a general rule, spiking will be performed at approximately 10 to 20 times the MDL unless other factors preclude that choice.

Several methods have established multi-laboratory performance based control limits for spike recovery of specific matrices (water and soil) through the USEPA. While Table 5.1 contains actual EMS Heritage developed control limits, the control limits used will be those determined by the USEPA for those equivalent analyses (assumes similar spiking levels used). Surrogate matrix spikes will be treated in the same manner for organic analyses. Refer to the specific methods for further explanation.

12.5.3.2 The spike (%R) recovery QC Acceptance Criteria for those organic methods providing specific limits are as found in Table 12.1, Organic Spike Recovery Criteria.**

12.5.3.3 The spike recovery (%R) QC Acceptance Criteria for Chromium VI analysis must be 100% ± 20%.

* Only the page pertaining to Chromium VI analysis is included.
** Not included since it does not pertain to Chromium VI analysis.

12.5.3.4 Post-digestion spikes (SPI01) are primarily used in metals analyses and are also referred to as the Method of Standard Additions (MSA). Two versions of this MSA are referenced in SW-846, Third Edition. The full method of standard additions generates a 4 point curve from which final results can be calculated. Another version of MSA is the Single-Point MSA, which essentially is a sample and matrix specific spike recovery.

The spike recovery criteria for a post-digestion spike is as follows:

SPI01 Criteria: 85-115% *Recovery*

12.5.4 Duplicate Matrix Spikes (DPS01, DPS02)

Duplicate matrix spikes measure both accuracy (see previous section for matrix spikes) and precision. As with matrix spikes, USEPA has established control limits for precision of specified methods for waters and soils. Those control limits are given as Relative Percent Difference (RPD) and will be used as control limits by EMS Heritage when applicable. When the methods do not specify RPD control limits, Table 5.1 of this QAP will be utilized when a sufficient number of observations allow for usable statistical limits. A maximum of 50 percent RPD will be allowed for organic testing and 20 percent RPD for inorganic testing. RPD's exceeding these limits will be considered as out-of-control for Duplicate Matrix Spike analyses.

Maximum Acceptable RPD
RPD Inorganic Criteria: 0-20
RPD Organic Criteria: 0-50

12.5.4.1 The Precision Control Limits for those organic methods providing specific limits are given in Table 12.3, Organic Precision Control Limits.*

12.5.5 Reagent Water or Reagent Matrix Spikes (LCS, DLCS)

Reagent water or reagent matrix spikes will be used as additional QC checks to monitor the effectiveness of the method. These QC types will utilize either reagent water spiked w/analyte (also known as Laboratory Fortified Blank) or a blank matrix (water, soil, sand, oil) which is known to exhibit minimal or no matrix interferences. EMS Heritage uses the acronyms of LCS (Laboratory Control Sample), DLCS (Duplicate Laboratory Control Sample) and/or ICV02 (Initial Calibration Verification - Prepped Standard) to identify the variations of these check samples. Analysis of this QC type will demonstrate that the method is in control when the Matrix Spike/Matrix Spike Duplicate (MS/MSD) recoveries are out-of-control. These QC types must be extracted/digested (if applicable) in the same preparation batch as the samples failing to meet QC criteria. Analysis may be optional for some organic tests when QC criteria for MS/MSD samples are met.

12.5.5.1 The reagent water or reagent matrix spikes (Laboratory Control Sample) are entered into the QCTS in LIMS as are all QC types. In addition, these QC samples are kept on control charts at the bench level. The minimum QC criteria to be utilized for these QC types will be the same criteria as for matrix specific precision and accuracy. More stringent criteria will be utilized where available. As a general rule the following criteria will apply for the Laboratory Control sample:

(LSC, DLCS, ICV02): 80-120% *Recovery (Accuracy)*
DLCS: 20% *RPD*

For Chromium VI analysis, the recovery must be 100% ± 10%.

* Not included since it does not pertain to Chromium VI analysis.

12.5.6 Quality Control Check Samples - Performance Evaluation Samples (P.E. Samples)

Acceptance criteria for P.E. samples are set by the organization running the study (EPA, commercial vendors). All results of P.E. samples are summarized to management.

12.5.7 Quality Control Check Standards or Initial and Continuing Calibration Check Standards (ICV01, CCV)

For Chromium VI analysis, the check standard (VER01) must have a mid-range recovery of 100% ± 10%.

12.5.8 Duplicate Samples (DUP01, DUP02)

Control limits for duplicate samples are identical to those set for duplicate matrix spike samples
, with one exception. Since duplicate precision is measured from analyte already present in the sample (no spike added) the level may not be detectable or may be very close to the detection limit. Concentrations of analyte less than 10 times the reporting detection limit (MDL, PQL, etc.) present in non-spiked (duplicate) samples will not be considered as out-of-control based on the QCTS data, but will be allowed a maximum RPD of 20 percent for inorganic analyses and 50 percent for organic analyses.

12.5.9 Methods Utilizing Internal Standards

The criteria which must be met for internal standard areas (when used) are found in Table 12.4, Internal Standard Area Criteria.*

12.5.10 Surrogate Recovery Criteria

Control limit criteria for methods which do not specify specific control limits will be developed from in-house data. EMS Heritage will not adopt criteria less stringent than the published method criteria. "Advisory" criteria will not require corrective action. Refer to Table 12.5, Surrogate Recovery Criteria* for method specific control criteria.

12.6 Review of Project Data

Project Managers are responsible for review of the overall project data before submission to the client. All laboratory "Certificates of Analysis" (Reports)

* Not included since it does not pertain to Chromium VI analysis.

and QA Summaries are reviewed and approved only by the QA Unit (see Reporting) prior to delivery to the Project Manager.

Project Managers are responsible for the following review prior to releasing the data report package to clients:

1. Review of supporting documentation including, but not limited to:

    a. Chain of Custody.

    b. Laboratory Analysis Request or Task Order Forms.

2. Review of all contract or project deliverables for completeness and accuracy including but not limited to:

    a. Reporting forms.

    b. Selected Raw data including chromatograms/ printouts.

    c. Adherence to specific DQO's.

    d. Analysis of all required QC Types at the required frequency.

3. Review of data for any obvious anomalous values, specifically those areas requiring knowledge of special project conditions possibly unknown to the QA Unit during review. Review large sets of data for comparability from one analytical batch or case to another in the project (QA Unit does not generally review an entire case at one time).

4. Adherence to reporting deadlines and shipment/delivery of report packages by the best means available to meet those deadlines.

12.7 Data Reporting

The Data Entry Supervisor is responsible for supervision of all data entry operators. The Data Entry (D.E.) Supervisor (or designee other than the original data entry operator) will verify 5 percent of all data entered into

LIMS. Locations having only one D.E. person will not check data in this manner; this responsibility will fall solely upon the QA Unit of that location.

Validation of data entry is performed for all data at a 2-5 percent frequency and is the responsibility of the QA Unit. The QA Unit may delegate this task to the QA Assistant or other staff person trained to perform that function.

The QA Unit is responsible for reviewing all final reports prior to release to clients. Reports are to be reviewed for:

1. Completeness - All parameters, detection limits, units, dates, descriptions, etc. must be complete and correct.

2. Consistency - All parameters must be internally consistent (CrVI ≤ Cr Total; TKN ≥ NH_3 - N, TS ≥ TDS, etc.)

3. Comparability - All parameters and units must be reported in such a way that data sets can be evaluated relative to each other as necessary.

12.7.1 All modifications to samples in the LIMS system must be documented on the Sample Modification Form, *Figure 12.2*. Original entries or additions (comments, etc.) to original entries will not utilize this form unless the sample is a "status 8" (released) category. All modifications to test codes must be approved by the Project Manager or the QA Unit. Modifications to samples that are completed and released (LIMS status 8) are possible only by the QA Unit staff. Any report that has been sent to a client and subsequently modified in any way by the QA Unit must be identified as an "amended report" and the date of amendment and the initials of the person making the amendment will be given on the certificate of analysis.

12.7.2 Samples of final reports and computer (LIMS) QC summaries are contained in the attached Final Report Examples.

12.7.3 Special Reporting Requirements

Sample analysis failing QC criteria due to matrix related interferences or samples requiring dilutions, etc. may have been analyzed 2 or more times. EMS Heritage will report a maximum of two analyses in those cases, or each analysis if client desires.

If a re-analysis of the sample fails the same criteria a second time and all other instrument criteria are in control, a matrix interference is assumed and both analyses may be reported. If only the reanalysis is in control, only the reanalysis will be reported.

Exceptions to this policy will be made for contract requirements and/or different client DQO's.

12.8 Data Storage

The following data is maintained for long term storage.

- bench sheets
- strip charts
- chromatograms, print-outs
- magnetic media used to store results
- invoices and financial statements
- project files
- customer data
- various raw data
- lab related periodicals

12.8.1 The records, hard copy and magnetic media, are kept on site for a period of six months to a year. As necessary, records may be transferred to an off-site records storage facility. The records storage facility must provide secure, access controlled and environmentally controlled storage of records. For all GLP (40 CFR, part 160) projects, records will be kept on-site.

After the completion of the requested work, records of the raw data, quality assurance data and reports will be kept by EMS for at least five (5) years unless otherwise required by law or regulations. Magnetic tapes are maintained, however, magnetic tapes have a storage life expectancy of less than ten (10) years.

12.8.2 The following is an overview of how records are stored and retrieved at EMS Heritage.

12.8.2.1 Storing Records

To send a box off site to a records storage facility the following three steps will be followed.

1. A box is filled with material of a related nature. The box is then marked to show the contents and the related service group and destruction date.

2. The box is then taken to the off-site storage coordinator where the box is given a unique number. The unique number, the contents, the associated service group, and the date stored are recorded in a ledger. Once the box is entered into the ledger it is scheduled to be picked up or delivered to the off-site records storage facility.

3. The off-site records storage facility stores the box and gives it a unique location code. Once the location code is assigned off-site records storage facility contacts the off-site storage coordinator, who enters the location code into the box's ledger entry. This allows for the recall of individual boxes.

12.8.2.2 Recalling Records

To recall records from off-site storage requires the EMS box number and the records storage facility location code, both of which are kept in a records storage ledger. A copy of the ledger is kept by the off-site storage coordinator, who can also recall the box from storage.

12.8.3 The LIMS system located in Indianapolis is backed up on a three cycle program. Every week a backup of the system is done. Weekly backup tape sets are rotated every week. So, if the computer is backed-up with tapes set "A" this week tape set "B" is used the next week. The tape sets are rotated back and forth every other week, except the last week of the month. For the last week of the month tape set "C" is used. By using this method it is possible to restore the

computer from as far back as one month. The tape sets are held at off-site storage, except when called back to do the backup. EMS Heritage also maintains over a month of "redo's" on tape that are backed up each day. "Redo's" are copies of the transactions needed to apply to an old backup of the database to bring it up to date. EMS Heritage also maintains a two week rotation of backups for the software. The software is backed up each day as well.

12.8.4 Data is not stored or organized per specific project unless contract or other client requirements dictate such. If storage by project is required, all raw data and final reports are copied and stored in a "central project file".

All original raw data and benchsheets are organized and stored in folders chronologically according to analytical batch and date of analysis.

00: APR THRU SEP

TABLE 5-

Method	Matrix	Parameter	N	% REC	N	RPD	Limit	Units
SW846-7060	Non-Spec Water	ARSENIC	106	68 - 127	56	0 - 16		
	Groundwater	ARSENIC	42	74 - 128	7	0 - 18		
	Oil	ARSENIC	19	36 - 130	3	0 - 4		
	S/S/S	ARSENIC	62	49 - 138	56	0 - 23		
SW846-7080	Non-Spec Water	BARIUM	2	70 - 109	1	0 - 6		
	S/S/S	BARIUM	10	59 - 136	58	0 - 13		
SW846-7090	S/S/S	BERYLLIUM	2	94 - 110				
SW846-7091	S/S/S	BERYLLIUM	4	44 - 162	3	0 - 20		
SW846-7130	Non-Spec Water	CADMIUM	1	85 - 85				
	Oil	CADMIUM	3	52 - 121	1	0 - 0		
	S/S/S	CADMIUM	10	76 - 118	34	0 - 18		
SW846-7131	Non-Spec Water	CADMIUM	5	69 - 139	1	0 - 15		
	Groundwater	CADMIUM	57	51 - 141	6	0 - 23		
	Oil	CADMIUM	5	56 - 134				
	S/S/S	CADMIUM	5	45 - 138	7	0 - 11		
SW846-7190	Non-Spec Water	CHROMIUM	2	82 - 111	4	0 - 14		
	Oil	CHROMIUM	3	84 - 136	1	0 - 0		
	S/S/S	CHROMIUM	18	63 - 168	57	0 - 24		
SW846-7191	Groundwater	CHROMIUM	2	77 - 124				
SW846-7196	Non-Spec Water	HEXAVALENT CHROMIUM	3	91 - 113				
	Groundwater	HEXAVALENT CHROMIUM	2	87 - 113				
SW846-7210	Non-Spec Water	COPPER	1	98 - 98				
	Groundwater	COPPER	1	88 - 88				

N = Sample count
REC = Percent Recovery (observed/actual*100)
RPD = Relative Percent Difference (2*(rep1-rep2)/(rep1+rep2)*100)

(qaqc.sql)

Table 11.5
ANALYTICAL RUN REQUIREMENT/FREQUENCY

Test	Method Reference	Instrumental QC							Prep. QC							
		ICV02	BLA01	CCV[a]	CDL01	DPS01[b]	DUP01[b]	SPI01 (or MSA)[c]	BLA02	(CLP) BLA02 SPI02	ICV02 or LCS[d]	(CLP) LCS/ SPI01	SPI02[b]	DPS02[b]	DUP02[b]	DLCS
Dissolved Solids	EPA	R, 10%	R, 5%	NA	NA	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	NA	NA	NA	5%	-
ISE Fluoride	EPA	C	B,E 10%	B,E 10%	B	P, 5%	P, 5%	P, 5%	P, 5%	-	P, 5%	-	P, 5%	P, 5%	P, 5%	-
Formaldehyde	AOAC						R, 10%				, 5%					
Gross Alpha, Beta (Radioactivity)	EPA	NA	B,E 20%	B	-	-	R, 10%	-	NA	NA	-	-	NA	NA	NA	-
Titrimetric Hardness	EPA	R	B,E 10%	NA	-	-	R, 10%	- -	NA	NA	R, 5%	-	NA	NA	NA	-
Hexavalent Chromium	SM	R	B,E 10%	B,E 10%	B	R, 10%	R, 10%	R, 10%	P, 10%	-	R, 5%	-	R, 10%	R, 10%	R, 10%	-
Hexavalent Cr	SW	R	B,E 10%	B,E 10%	B	R, 10%	R, 10%	R, 10%	P, 10%	-	R, 5%	-	R, 10%	R, 10%	R, 10%	-
Iodine	SM	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
TKN	EPA	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
Nitrate-N (Brucine)	EPA	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
Nitrate-N (Colorimetric)	EPA	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
Nitrite-N	EPA	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
Nitrate-Nitrite-N	EPA	C	B,E 10%	B,E 10%	-	R, 10%	R, 10%	R, 10%	NA	NA	R, 5%	-	NA	NA	NA	-
Nuisance Dust	NIOSH	R	R	B,E 10%	NA	NA	R, 10%	NA	NA	NA	NA	NA	NA	NA	NA	-
Oil & Grease	EPA413.1	R	R	NA	-	-	R, 10%	-	NA	NA	-	-	NA	NA	NA	-
Oil & Grease	SW 9071	R	R	NA	-	-	R, 10%	-	NA	NA	R, 5%	-	NA	NA	NA	-

FIGURE 12.1
EMS LABORATORIES

DATA REVIEW PROTOCOL

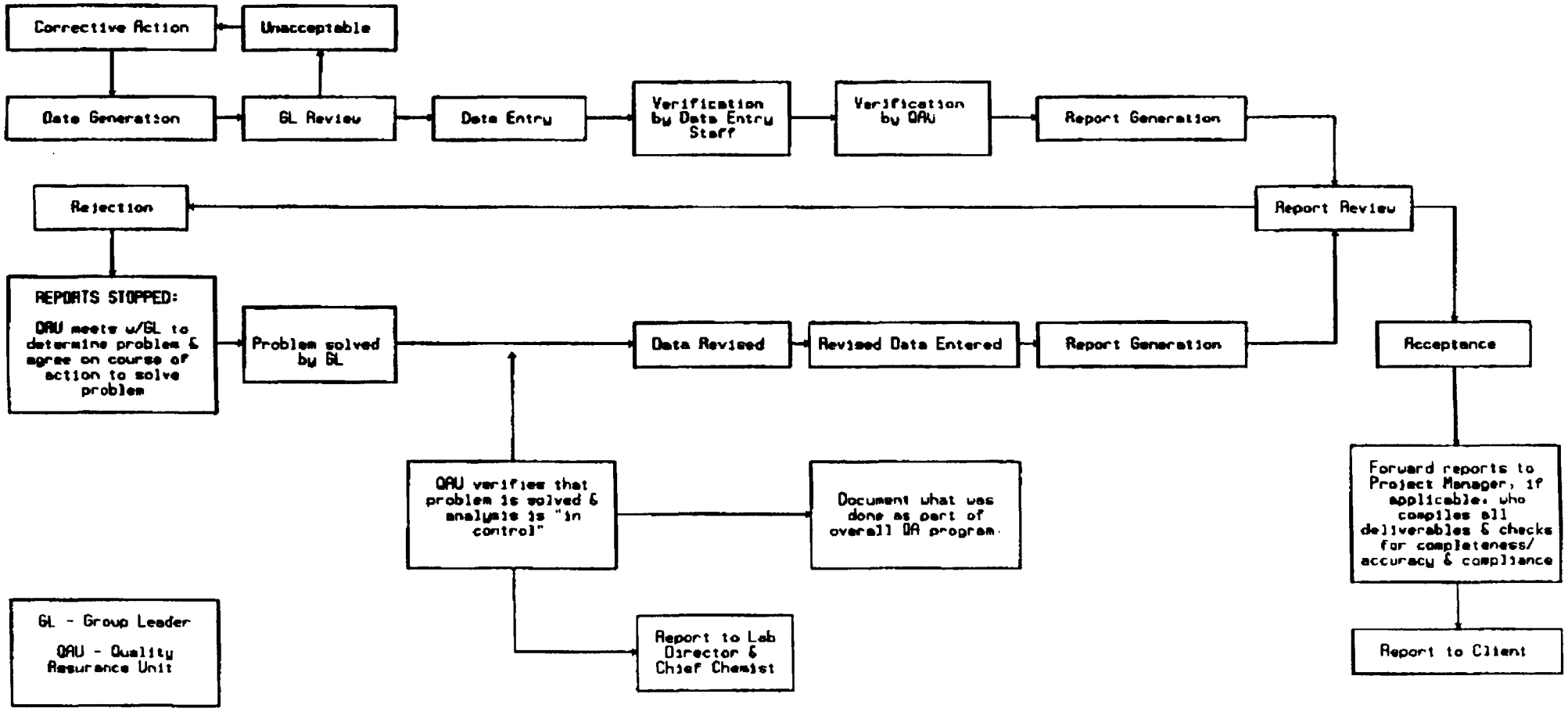


FIGURE 12.2

SAMPLE MODIFICATION FORM

REQUEST FROM:_____ DATE_____

SAMPLE STATUS:_____

SAMPLE NUMBERS:_____

_____

CLIENT:_____

TEST CODE ADDED/CANCELLED:_____

TEST CODE CHANGED FROM _____ TO _____

RESULTS MODIFICATIONS:_____

_____

_____

_____

REASON FOR ADDITION OR CHANGE:_____

_____

_____

_____

MODIFIED BY:_____ DATE _____

PROJECT MANAGER'S APPROVAL INITIALS:_____ DATE _____

ACCOUNT MANAGER'S APPROVAL INITIALS:_____ DATE _____

OLD PRICE:_____ NEW PRICE:_____

FINAL REPORT EXAMPLES

CERTIFICATE OF ANALYSIS

Service Location	Received	Lab ID
EMS HERITAGE LABORATORIES, INC. 7901 W. MORRIS ST. INDIANAPOLIS, IN 46231 (317)243-8305	23-APR-91	A228370
	Complete 05-JUN-91	PO Number 90607684-59A
	Printed 13-JUN-91	Sampled

Report To

Bill To

Sample Description

FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Analyst: J. CARSON Analysis Date: 30-APR-91 Test: P130.4. 0

Parameter	Result	Det. Limit	Units
INITIAL WEIGHT OR VOLUME	50		mL
FINAL WEIGHT OR VOLUME	50		mL

BARIUM ICP SW846-6010

Analyst: M. JAO Analysis Date: 01-MAY-91 Instrument: ICP Test: M104.3. 0

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Parameter	Result	Det. Limit	Units
BARIUM	0.049	0.010	mg/L

CADMIUM ICP SW846-6010

Analyst: M. JAO Analysis Date: 01-MAY-91 Instrument: ICP Test: M108.3. 0

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Parameter	Result	Det. Limit	Units
CADMIUM	BDL	0.0050	mg/L

CHROMIUM ICP SW846-6010

Analyst: M. JAO Analysis Date: 01-MAY-91 Instrument: ICP Test: M110.3. 0

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Parameter	Result	Det. Limit	Units
CHROMIUM	BDL	0.010	mg/L

NICKEL ICP SW846-6010

Analyst: M. JAO Analysis Date: 01-MAY-91 Instrument: ICP Test: M122.3. 0

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Parameter	Result	Det. Limit	Units
NICKEL	BDL	0.010	mg/L

SILVER ICP SW846-6010

Analyst: M. JAO Analysis Date: 01-MAY-91 Instrument: ICP Test: M130.3. 0

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Parameter	Result	Det. Limit	Units
SILVER	BDL	0.010	mg/L

QUALITY ASSURANCE REPORT

Service Location	Received	Lab ID
EMS HERITAGE LABORATORIES, INC. 7901 W. MORRIS ST. INDIANAPOLIS, IN 46231 (317)243-8305	23-APR-91	A228370
	Complete	PO Number
	05-JUN-91	90607684-59A
	Printed	Sampled
	13-JUN-91	

Sample Description

BARIUM ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M104.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

QC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
I 01	Q239253	WP1083	BARIUM	2.00		2.06	mg/L	103	
DPS02	Q231974	A228372	BARIUM	0	2.00	1.92	mg/L	96	4
SPI02	Q231973	A228372	BARIUM	0	2.00	1.84	mg/L	92	
C	Q239277	INORGANIC	BARIUM	5.00		4.52	mg/L	90	
B 01	Q239276	NA	BARIUM			< 0.004	mg/L		
LCS	Q231971	NA	BARIUM	20.0		18.9	mg/L	94	
B 02	Q231972	NA	BARIUM			< 0.004	mg/L		
S PLE	A228370		See Certificate of Analysis						
CCV	Q239275	INORGANIC	BARIUM	5.00		4.81	mg/L	96	
BLA01	Q239274	NA	BARIUM			< 0.004	mg/L		

CADMIUM ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M108.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

QC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
01	Q239254	WP1083	CADMIUM	0.400		0.425	mg/L	106	
01	Q239259	WP1083	CADMIUM	0.400		0.407	mg/L	102	
DPS02	Q231974	A228372	CADMIUM	0	0.050	0.0415	mg/L	83	4
SPI02	Q231973	A228372	CADMIUM	0	0.050	0.0398	mg/L	80	
01	Q239279	NA	CADMIUM	0.0100		0.0116	mg/L	116	
CCV	Q239277	INORGANIC	CADMIUM	5.00		4.48	mg/L	90	
BLA01	Q239276	NA	CADMIUM			< 0.002	mg/L		
	Q231971	NA	CADMIUM	0.500		0.407	mg/L	81	
02	Q231972	NA	CADMIUM			< 0.002	mg/L		
SAMPLE	A228370		See Certificate of Analysis						
	Q239275	INORGANIC	CADMIUM	5.00		4.80	mg/L	96	
01	Q239274	NA	CADMIUM			< 0.002	mg/L		
DL01	Q239270	NA	CADMIUM	0.0100		0.0122	mg/L	122	

CHROMIUM ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M110.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

QC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
CV01	Q239254	WP1083	CHROMIUM	0.400		0.421	mg/L	105	
01	Q239259	WP1083	CHROMIUM	0.400		0.400	mg/L	100	
02	Q231974	A228372	CHROMIUM	0	0.200	0.193	mg/L	97	3
SU2	Q231973	A228372	CHROMIUM	0	0.200	0.188	mg/L	94	
L01	Q239279	NA	CHROMIUM	0.0200		0.0260	mg/L	130	

EMS HERITAGE LABORATORIES, INC.

Lab Sample ID: A228370

QC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
V	Q239277	INORGANIC	CHROMIUM	5.00		4.54	mg/L	91	
A01	Q239276	NA	CHROMIUM			< 0.004	mg/L		
LCS	Q231971	NA	CHROMIUM	2.00		1.85	mg/L	92	
A02	Q231972	NA	CHROMIUM			< 0.004	mg/L		
MPLE	A228370		See Certificate of Analysis						
CCV	Q239275	INORGANIC	CHROMIUM	5.00		4.83	mg/L	97	
A01	Q239274	NA	CHROMIUM			< 0.004	mg/L		
L01	Q239270	NA	CHROMIUM	0.0200		0.0210	mg/L	105	

NICKEL ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M122.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
V01	Q239254	WP1083	NICKEL	0.400		0.419	mg/L	105	
ICV01	Q239259	WP1083	NICKEL	0.400		0.408	mg/L	102	
PS02	Q231974	A228372	NICKEL	0	0.500	0.468	mg/L	94	3
102	Q231973	A228372	NICKEL	0	0.500	0.457	mg/L	91	
CL01	Q239279	NA	NICKEL	0.0200		0.0229	mg/L	115	
CCV	Q239277	INORGANIC	NICKEL	5.00		4.50	mg/L	90	
A01	Q239276	NA	NICKEL			< 0.004	mg/L		
S	Q231971	NA	NICKEL	5.00		4.62	mg/L	92	
BLA02	Q231972	NA	NICKEL			< 0.004	mg/L		
MPLE	A228370		See Certificate of Analysis						
V	Q239275	INORGANIC	NICKEL	5.00		4.79	mg/L	96	
BLA01	Q239274	NA	NICKEL			< 0.004	mg/L		
CL01	Q239270	NA	NICKEL	0.0200		0.0199	mg/L	99	

SILVER ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M130.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

QC Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
V01	Q239253	WP1083	SILVER	2.00		2.05	mg/L	102	
S02	Q231974	A228372	SILVER	0	0.050	0.0492	mg/L	98	2
SP102	Q231973	A228372	SILVER	0	0.050	0.0479	mg/L	96	
CL01	Q239252	NA	SILVER	0.0200		0.0200	mg/L	100	
V	Q239277	INORGANIC	SILVER	1.00		0.912	mg/L	91	
A01	Q239276	NA	SILVER			< 0.004	mg/L		
LCS	Q231971	NA	SILVER	0.500		0.468	mg/L	94	
A02	Q231972	NA	SILVER			< 0.004	mg/L		
MPLE	A228370		See Certificate of Analysis						
CCV	Q239275	INORGANIC	SILVER	1.00		0.950	mg/L	95	
A01	Q239274	NA	SILVER			< 0.004	mg/L		
L01	Q239271	NA	SILVER	0.0200		0.0208	mg/L	104	

IRON ICP SW846-6010

Analyst : M. JAO — Analysis Date: 01-MAY-91 — Instrument: ICP — Test: M115.3.0

Reviewer: S. ENDERSEN — Review Date: 02-MAY-91 — File ID: — Run: R119838

Prep: FAA OR ICP ACID DIGESTION OF AQUEOUS SAMPLES SW846-3005

Type	Identifier	Source	Parameter	True/Sampl	Spike Val	Observed	Units	% Rec	RPD
01	Q239254	WP1083	IRON	0.400		0.425	mg/L	106	
ICV01	Q239259	WP1083	IRON	0.400		0.413	mg/L	103	
PS02	Q231974	A228372	IRON	0	1.00	0.968	mg/L	97	3
02	Q231973	A228372	IRON	0	1.00	0.942	mg/L	94	
CL01	Q239279	NA	IRON	0.0400		0.0407	mg/L	102	
CCV	Q239277	INORGANIC	IRON	5.00		4.59	mg/L	92	

APPENDIX B.6

EMS HERITAGE LABORATORIES, INC.

PERFORMANCE AND SYSTEMS AUDITS

14.0 Performance and Systems Audits

14.1 Performance Audits

A performance audit is an independent check to evaluate the data produced by a laboratory's analytical system, and may be categorized as a quantitative appraisal of quality. There are several ways that this is done:

1. Worksheet review
2. Oral worksheet review
3. On-site analyst work review
4. Independent or check sample review
5. Intra and interlaboratory check sample, or proficiency test (performance evaluation) sample analysis review

14.1.1 Internal Performance Audits

Internal performance audit worksheet reviews are conducted by the QA Officer and/or the QA Unit. These reviews must be conducted at a frequency necessary to assure the accuracy of either the total measurement system or its component parts. Worksheet reviews by the QAO and/or QAU will be on-going but will occur at a minimum frequency of semi-annually for selected tests. This will be in addition to (or will be a more in-depth review of all data records) the 2-5 percent frequency of data entry validation performed by the QAU.

Internal performance audit worksheet reviews will consist of evaluations of all data and related supporting documentation to assure that all required QC checks are being made and evaluation criteria followed. Reports relating to internal performance audit worksheet reviews are confidential and will be released only upon approval from the President of EMS Heritage.

14.1.2 Performance Audit-Proficiency Test Samples

Performance Audits at EMS Heritage also consist of analysis of independent or commercial check samples and participation in performance evaluation sample programs.

Refer to table 14.0, Reference Materials And Performance Evaluation ~~Sample Programs~~ Participation for the sources of reference materials used and the on-going performance audit participation.

All information generated from Performance Evaluation (P.E.) sample programs will be made available during systems audits or upon request. Blind samples and split samples may be submitted as deemed necessary by the QAO or the QAU.

14.2 Systems Audits

A systems audit is an on-site inspection and review of a laboratory's quality control system and may be categorized as a qualitative appraisal of quality. It will cover any or all of the operational quality control elements of the quality assurance program. Systems audits include but are not limited to the following:

1. Sample handling: receiving, custody, log-in, storage
2. Sample analysis: written SOPs and analytical methods, protocols
3. Records control
4. Documentation: bound notebooks where required; records of all sample handling and analytical procedures
5. Preventative maintenance: adequate records, procedures.
6. Proficiency testing
7. Personnel practices
8. Training
9. Workload
10. Instrumentation and facilities

14.2.1 External Systems Audits

Systems audits are performed frequently at EMS Heritage by State and Federal agencies as part of the participation in sample analyses for

TABLE 14.~~0~~1
REFERENCE MATERIALS AND PERFORMANCE EVALUATION PARTICIPATION

Source	Type	Frequency	Parameters	Division
APG	Unknowns	Monthly	All	All
EPA-WP	Unknowns	Semi-Annually	All	All
EPA-WS	Unknowns	Semi-Annually	All	All
North Carolina-Wastewater	Unknowns	Semi-Annually	Metals, General Chemistries	Charlotte
State of New York	Unknown	Quarterly	All	Indianapolis
State of New Jersey	Unknown	Semi-Annually	All	Indianapolis
EPA Reference	Known	On-Going	All	All
APG	Known	On-Going	All	All
NIST	Known	On-Going		All
EPA QB-Inorganics	Unknown	Quarterly	Metals	Indianapolis
EPA QB-Organics	Unknown	Quarterly	Organics	Indianapolis
Chemical Waste Mgmt.	Unknown	Quarterly	All	Indianapolis, Chicago

governmental organizations. In addition, many of our commercial clients perform routine audits of EMS Heritage. Evaluation by other appropriate outside experts is to be performed in the event that regulatory personnel are not available. At a minimum, systems audits will occur annually. EMS Heritage Laboratories will welcome any external audit from an organization which currently or proposes to submit samples for analysis.

14.2.2 Internal Systems Audits

In order to verify that each laboratory division is performing according to the standards established by EMS Heritage Laboratories, Inc., an internal audit system has been established. Internal systems audits will be conducted no less frequently than annually. The auditor will be appointed by and directly responsible to the President of the corporation.

The President may appoint for any given audit the following staff:

1. Quality Assurance Officer
2. Quality Assurance Unit
3. Outside (Contracted) Auditor

EMS Heritage also encourages the individual groups at each division to perform a self-audit at periodic intervals to assess their overall operation.

These audits will be scheduled by the auditor and each laboratory director. All reports relating to internal systems audits will be treated as an internal document only; release to any outside entity will be only upon approval of the President of EMS Heritage.

To facilitate the procedure and make it consistent from division to division, a set of audit procedures and protocols has been developed and will be used consistently throughout the corporation. These forms will be used as a guideline and a tool for documentation of problems and to assure that all important areas are covered. The forms are not an end in themselves and will be subject to change/improvement by the QAO at his discretion in order to facilitate and improve the auditing process. Refer to the attached Internal Systems Audit Forms.

14.2.3 Internal Systems Audits - Procedures and Protocols

Not all areas (service groups) will necessarily be audited during each visit. During each audit, at least one analytical run will be randomly selected from the service group(s) chosen. This run will be thoroughly checked using the audit checklist forms and any other pertinent audit questions.

Upon completion of the audit, the auditor will conduct an exit interview with the QA Unit and the Laboratory Director and any other personnel deemed appropriate. A copy of the completed audit forms as well as any supporting narrative will be given to the QA Unit and the Laboratory Director at that time or immediately upon completion. A copy of all of these materials will also be sent to the President, Vice President of Operations, Quality Assurance Officer and all QA Units of all EMS Divisions. This sharing of audit information with all divisions is to provide not only a report of findings but will also serve to point out potential deficiencies which may exist at the respective divisions not audited. The auditor will retain a copy of the materials to use in the next audit. The QAO will confer with the President to prioritize items for the next audit.

Generally the audit will consist of the auditor reviewing with the group leader and the analyst all the steps involved in generating a run of reportable data. It will also include an overview of the entire lab, address issues relevant to the operation as a whole (i.e. cooler logs, log-in procedures, oven logs, etc.) Any deficiencies will be noted on the forms. Deficiencies will be discussed at the time they are noted and the auditor will explain what will be required to correct this deficiency.

Form 1 will always be ~~filled out~~ completed. It addresses general laboratory practices. Form 2 will be ~~filled out~~ completed during each audit for each service group audited. Forms 3-5 will be used on a service group specific basis only.

INTERNAL SYSTEMS AUDIT FORMS

FORM 1

DATE _____

LOCATION _____

AUDITOR _____

GENERAL INFORMATION

		YES	NO	COMMENTS
1)	Does the laboratory have copies of SOPs/methods manuals and QA Plans available to all personnel?			
2)	Are training records maintained and up to date? Is training by group leader or above?			
3)	Is a Sample Custodian designated?			
4)	Are written Standard Operating Procedures (SOPs) developed for receipt and storage of samples?			
5)	Are samples stored in such a way that their preservation is maintained?			
6)	Are refrigerator/freezer logs maintained, checked daily and up to date?			
7)	Are excursions in cooler temperature noted and appropriate actions taken as required?			
8)	Are volatiles stored separately from semi-vols?			
9)	Does someone responsible review and initial the sample log and log sheets daily?			
10)	Are contamination free areas provided for trace level analytical work?			
11)	Does the facility appear clean and safe?			
12)	Are toxics handled in fume hoods? Are fume hoods checked & documented quarterly?			
13)	Are the toxic chemical handling areas either a stainless steel bench or an impervious material covered with absorbent material?			

PAGE 2 OF FORM 1

		YES	NO	COMMENTS
14)	Is there documented "trace-free" water available for preparing standards and blanks?			
15)	Is the conductivity of "trace-free" water routinely checked and recorded?			
16)	Is (Are) the analytical balance(s) correctly located (free from drafts and rapid temperature changes) and checked semi-annually by a certified technician and documented?			
17)	Are the balances checked with class S weights and documented at least weekly?			
18)	Are solvent storage cabinets properly vented in order to prevent possible laboratory contamination?			
19)	Are reagent grade (or higher purity) chemicals used to prepare standards and reagents?			
20)	Are reagents dated upon receipt and upon opening? Is First-in, First-out method used?			
21)	Are reagents standardized before use?			
22)	Generally, are reference materials properly labeled with concentrations, preparation date, solvent, preservative expiration date and name of person preparing?			
23)	Are spike/calibration stock standards preparation and tracking logbook(s) maintained?			
24)	Are the primary standards approved by the EMS QAP?			
25)	Are bench sheets filed so that they are readily accessible?			
26)	Are standards and samples (& extracts) stored separately?			
27)	Are samples checked for proper preservation upon arrival? Is this noted within LIMS?			
28)	Are logs maintained for all ovens and incubators?			
29)	Is an NBS thermometer available?			

PAGE 3 OF FORM 1

		YES	NO	COMMENTS
30)	Are copies of EPA - PEs and others such as APG on file?			
31)	Is the distilled/deionized water system functioning properly? Is it checked daily and noted in a logbook?			
32)	Are samples requiring chain of custody documentation properly checked before signing chain of custody papers?			
33)	What is done with lab waste? _____ _____ _____			
34)	Are chemical waste disposal policies/procedures adequate?			
35)	Are blind samples introduced into the lab by the QAO?			
36)	Are corrections on bench sheets, lab books, etc. made in such a way that initial entries are legible?			
37)	Are such corrections initialed and dated?			

FORM 2

DATE_____

SERVICE GROUP _____ AUDITOR _____

LOCATION _____ ANALYST _____

	TEST	YES	NO	COMMENTS
1)	Are methods manuals & QAP's available to the analysts?			
2)	Is the SOP for glassware washing posted at the cleaning stations?			
3)	Are the types and numbers of required Blanks being checked? Bla 01? Bla 02? Cal 00?			
4)	Are blank data logged to the QA sheet and transferred to LIMS?			
4a)	Does blank data appear to be "in control"?			
4b)	Do original instrument outputs agree with what was reported?			
5)	Are calibration curves maintained for all analytes?			
6)	Are calibrations verified (1 point) or performed (5 points) prior to the analysis of samples CAL01 + CAL00? Comment: _____			
7)	Is an EPA, NIST or other approved external reference used to check and verify concentrations of CAL standards and is the result recorded on the QA sheet?			
7a)	Are the results recorded in LIMS?			
8)	Do the analysts record bench data in a neat and accurate manner?			

PAGE 2 OF FORM 2

		YES	NO	COMMENTS
8a)	Is all quality assurance data correctly transferred from the Bench/QA sheets into LIMS?			
9)	Is a calibration verification performed at the correct frequency?			
9a)	Documented to LIMS?			
10)	Is a prepped performance check (LCS) analyzed once per run or as required by the QAP? (eg one per set of 10)			
11)	Is a prepped sample spike (SP102) analyzed with each run or as required by the QAP? (eg one per set of 10)			
12)	Is a prepped sample replicate analyzed (or a duplicate spike?) at the appropriate frequency according to the QAP? (eg one per set of 10)			
13)	If any problems occur, has corrective action been taken and documented?			
	Comments: _____			
14)	Is a calibration verification solution analyzed at the end of each run?			
15)	Is the method of internal standards used for GC/MS?			
16)	List internal standards used for VOA. (Attach copy of report)			
	Are these correct for the method?			
17)	List internal standards used for SVOA's. (attach copy of report)			
	Are these correct for the method?			
18)	Are surrogates properly analyzed and reported? Are surrogates within CLP acceptance ranges?			

PAGE 3 OF FORM 2

		YES	NO	COMMENTS
19)	Are standard recoveries correctly calculated? Are spike recoveries correctly calculated? Are duplicate recoveries correctly calculated? Are they all documented in LIMS?			
20)	Does a manual recalculation agree with concentrations otherwise calculated and/or reported? (ie verify electronic integrator performing and correct peaks were chosen for GC, GC/MS)			
21)	For GC/MS, are unknowns (non-target compounds) correctly searched in the NBS library and documented as present with an estimated concentration?			
22)	For ICP, are interelement interference check solutions properly analyzed and documented prior to each run?			
23)	Have detection limits been empirically determined according to 40CFR136 for the analytes determined?			
24)	Are standards correctly Labelled? Logged & documented?			
25)	Are instrument operating manuals readily available?			
26)	Are data acceptance criteria developed and used for Blanks? Duplicates? Verification standards? Spikes?			
27)	Are instrument maintenance logs in place and maintained?			
28)	Is the mercury analyzer operational and well maintained (i.e. properly vented)?			
29)	Is preventive maintenance applied and documented?			
30)	Are data calculations spot-checked by a second person? Does this person initial these checked calculations?			

PAGE 4 OF FORM 2

		YES	NO	COMMENTS
31)	Do supervisory personnel review bench sheets and initial them?			
32)	Do records indicate that corrective action was taken as necessary?			
33)	Has a cooperative attitude been displayed?			
34)	Has corrective action indicated during previous visits been implemented?			
35)	Are extractions/digestion performed within holding times?			
36)	Are analyses performed within holding times?			
37)	Are sample preparation methods correct?			
38)	Is a background corrector properly used? (AA, GFAA, ICP)			
39)	Is an approved methodology used for the runs being examined?			
40)	Have acceptance criteria for start-up QC been met?			
41)	Do instrument outputs (strip charts, print-outs, bench sheets, etc.) agree with what is in the final report?			
42)	Are labs data validation procedures adequate?			
43)	Based upon blank, standard, spike and duplicate data, are goals of data accuracy and precision being met?			
44)	Is the method of standard additions being used correctly for HGA analyses?			
45)	Is analytical sensitivity adequate?			
46)	Are instrument guidelines and/or acceptance criteria used at the bench and at data review which determine the acceptability and reportability of data?			
47)	Are Blanks carried through entire process?			
48)	Are desiccants changed and documented?			

PAGE 5 OF FORM 2

		YES	NO	COMMENTS
49)	Are TOC, TOX and BTU instrument logs current?			
50)	Are normalities of titrants routinely checked?			
51)	Are sufficient cyanide distillation apparatus available to routinely analyze all samples within the required holding period?			

FORM 3

LAB _____

DATE _____

AUDITOR _____

METALS GROUPS

		YES	NO	COMMENTS
1)	Are samples for mercury analysis collected in glass and preserved with HNO_3 and Permanganate?			
2)	Are mercury determinations performed within 28 days of sample collection?			
3)	Are proper sample digestion techniques used and documented?			
4)	Is background correction used a) For all HGA work? b) As required for flame AA work?			
5)	Are correct matrix modifiers used for HGA?			
6)	Is an ionization suppressor used as needed for flame AA?			
7)	For ICP are interelement corrections checked?			
8)	Do prep sets of 10 include proper QA and acceptance criteria?			
9)	Are wavelengths used recorded on bench sheets?			
10)	Are flame/furnace/ICP programs adequate? a) Integration times adequate? b) Furnace temperature programs correct? c) Sample aliquot or deposition times adequate?			
11)	Are sensitivity guidelines available at instruments?			

FORM 4

LAB _____

DATE _____

AUDITOR _____

GAS CHROMATOGRAPHY

		YES	NO	COMMENTS
1)	Are samples collected in amber glass bottle with teflon-lined lids?			
2)	Are samples kept at 4°C			
3)	Are samples extracted within 7 days?			
3a)	Analyzed within 40 days?			
4)	Are standards properly documented?			
5)	Are standards prepared fresh regularly?			
6)	Is there a well maintained log, documenting stability of all detectors?			
7)	Are Arochlor 1221 and 1232 standards run at the proper frequency and the data maintained for on-site inspection?			

FORM 5

LAB _____

DATE _____

AUDITOR _____

GC/MS

		YES	NO	COMMENTS
1)	Are samples extracted and/or analyzed within all required holding times?			
2)	Are control limits established and used for CCC and SPCC samples?			
3)	Is QA data properly transferred to LIMS?			
4)	Are Blanks, Spikes and Standards analyzed at the appropriate frequency?			
5)	Is Spike/spike duplicate data being generated?			
6)	Are surrogates properly analyzed with all samples?			
7)	Are surrogate results tabulated?			
8)	Are both 5 and 25 ml purge vessels available and properly used?			
9)	Are correct internal standards being used?			
10)	Is data reviewed prior to issuing final report?			
11)	Has QAO reviewed 2-5% of the runs?			
12)	Are reviewed bench sheets initialed?			
13)	Are all tuning criteria met?			
14)	Is the trap for the purge and trap filled with the proper adsorbent?			

PAGE 2 OF FORM 5

		YES	NO	COMMENTS
15)	Is raw data being archived and documented properly (i.e magnetic tape)?			
16)	Is a split/splitless capillary injector in place?			

APPENDIX C

LANCASTER LABORATORIES, INC.

QUALITY ASSURANCE PLAN

1. Laboratory Quality Assurance Plan

This document provides the laboratory portion of the response to EPA's "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" QAMS-005/80, Sections 5.1 - 5.16 as revised December 29, 1980.

As much as possible, the procedures in this document have been standardized to make them applicable to all types of environmental monitoring and measurement projects. However, under certain site specific conditions, all of the procedures discussed in this document may not be appropriate. In such cases it will be necessary to adapt the procedures to the specific conditions of the investigation.

Director of Quality Assurance _____

Section		Pages	Revision	Date
1.	Title Page	1		
2.	Table of Contents	1		
3.	Project Description	1		
4.	Project Organization and Responsibility	4		
5.	QA Objectives for Measurement Data, in terms of precision, accuracy, completeness representativeness and comparability	2		
6.	Sampling Procedures	2		
7.	Sample Custody	9		
8.	Calibration Procedures and Frequency	3		
9.	Analytical Procedures	46		
10.	Data Reduction, Validation and Reporting	5		
11.	Internal Quality Control Checks	4		
12.	Performance and Systems Audits	15		
13.	Preventive Maintenance	2		
14.	Specific Routine Procedures Used to Access Data Precision, Accuracy and Completeness	3		
15.	Corrective Action	3		
16.	Quality Assurance Reports to Management	1		
Appendix A - Reporting Forms		10		

3. Project Description

Tests will be performed according to the analytical methodology set forth in Section 9. These OSHA and NIOSH references provide specific analytical procedures to be used and define the specific application of these procedures. The soil vapor samples will be analyzed for selected Volatile Organic Compounds and Phenol. Proven instruments and techniques will be used to identify and measure the concentrations of all analytes. The laboratory will employ state-of-the-art procedures to perform all organic analyses, including all necessary preparation for analysis. The client is responsible for providing specifics on the project site.

4. Project Organization

The objectives of the laboratory Quality Assurance Program are to establish procedures which will ensure that data generated in the laboratory are within acceptable limits of accuracy and precision, to ensure that quality control measures are being carried out, and to ensure accountability of the data through sample and data management procedures. To this end, a Quality Assurance Department has been established. The Director of Quality Assurance reports directly to the President of the Laboratory and has no direct responsibilities for data production, thus avoiding any conflict of interest.

The attached organizational charts show the key personnel in both Corporate Services and the Environmental Sciences Division. Resumes of key individuals may be found in the enclosed Qualification Manual.

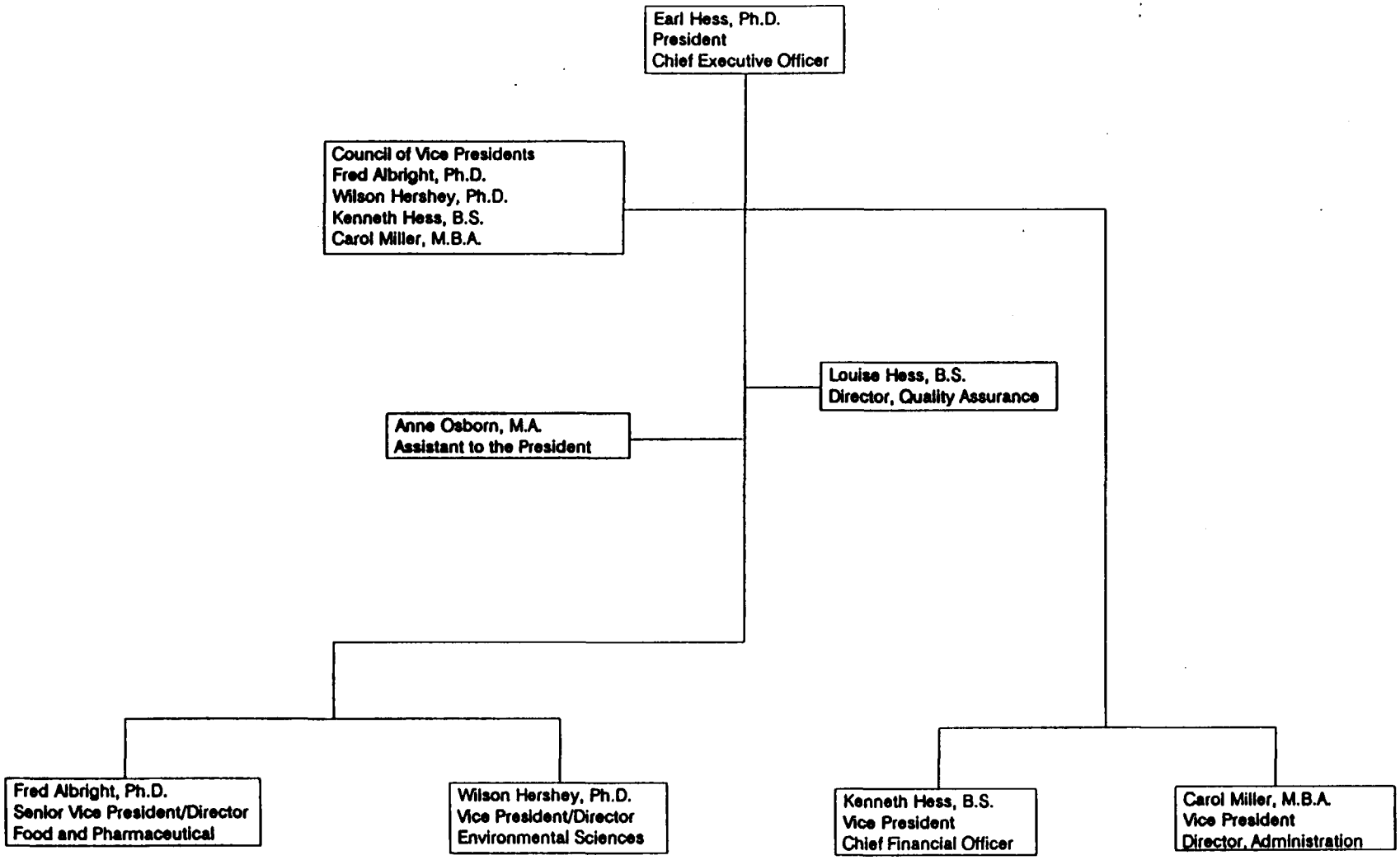
The Sample Administration Group will be responsible for receiving samples, signing the external chain-of-custody, checking sample condition, assigning unique laboratory sample identification numbers, assigning storage locations, checking and adjusting preservation, and homogenizing the sample as needed.

Group Leaders listed in each technical area are responsible for performing laboratory analyses, quality control as specified in the methods, instrument calibration, and technical data review. Data is reported using a computerized sample management system, which tracks sample progress through the laboratory and generates client reports when all analyses are complete. Quality control data is entered onto the same system for purposes of charting and monitoring data quality.

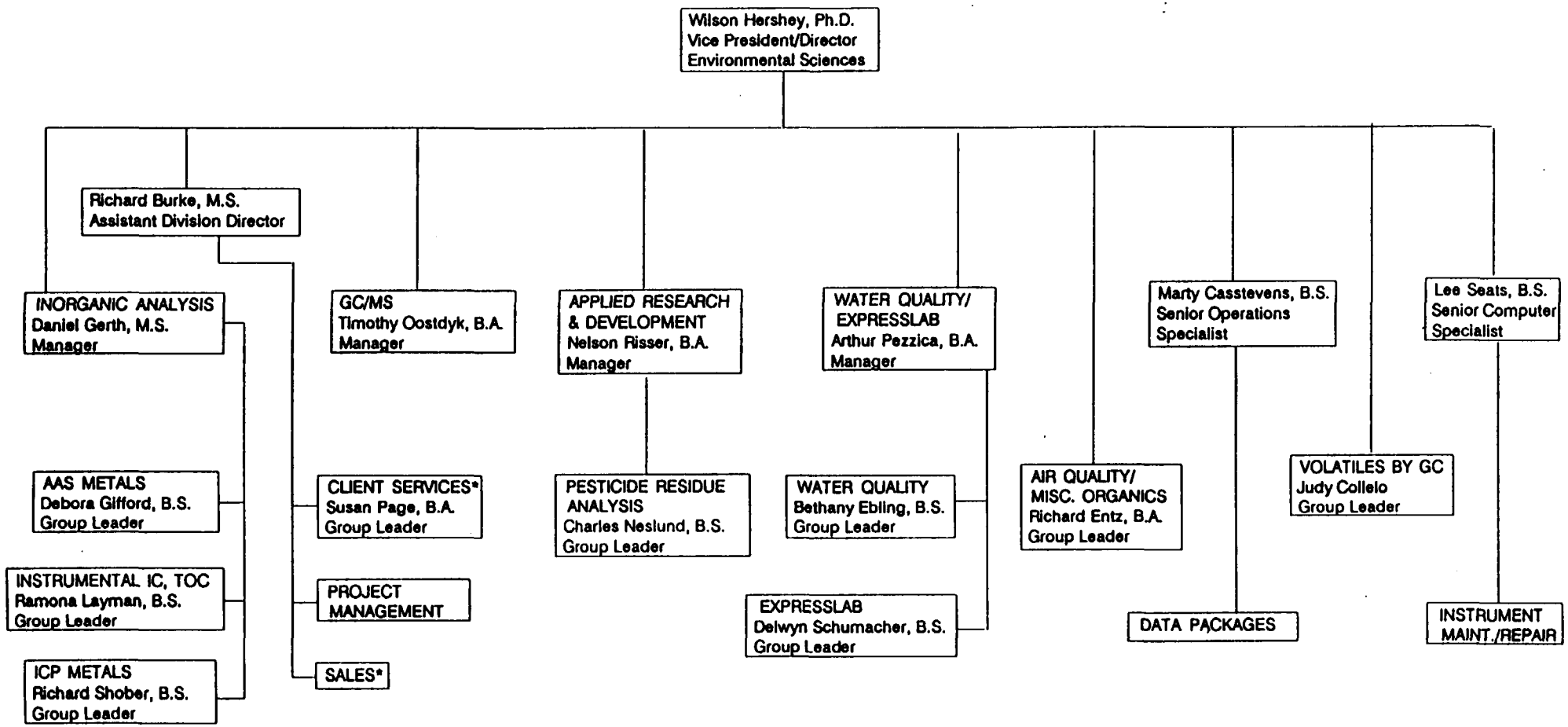
The Quality Assurance Department is responsible for reviewing quality control data, conducting audits in the laboratory and reporting findings to management, maintaining current copies of all analytical methods, maintaining copies of computer code used to calculate and report results, submitting blind samples to the laboratory and ensuring that appropriate corrective action is taken when quality problems are observed.

Data package deliverables are available upon request. The Quality Assurance Department reviews the contents of the deliverables for completeness and to be sure that all quality control checks were performed and met specifications. This step includes review of holding times, calibrations, instrument tuning, blank results, duplicate results, matrix spike results, and surrogate results. Every attempt to meet specifications will be made and any item outside of the specifications will be noted in the narrative. The laboratory will not validate data with regard to useability since this generally requires specific knowledge about the site.

Lancaster Laboratories, Inc.
Corporate Services



Lancaster Laboratories, Inc.
Environmental Sciences



*These groups cover client services and sales for all technical operations.

5. QA Objectives For Measurement Data

Quality Assurance is the overall program for assuring reliability of monitoring and measurement data. Quality control is the routine application of procedures for obtaining set standards of performance in the monitoring and measurement process. Data quality requirements are based on the intended use of the data, the measurement process, and the availability of resources. The quality of all data generated and processed during this investigation will be assessed for Precision, Accuracy, Representativeness, Comparability, and Completeness.

Precision - Precision is determined by measuring the agreement among individual measurements of the same property, under similar conditions. The laboratory objective is to equal or exceed the precision demonstrated for the applied analytical method on comparable samples. The degree of agreement is expressed as the relative percent difference (RPD%). Evaluation of the RPD% is based on statistical evaluation of past lab data for organic and inorganic analyses. External evaluation of precision is accomplished by analysis of Standard Reference Material and interlaboratory performance data.

Accuracy - Accuracy is a measure of the closeness of an individual measurement to the true or expected value. Analyzing a reference material of known concentration or reanalyzing a sample which has been spiked with a known concentration/amount is a way to determine accuracy. Accuracy is expressed as a percent recovery (%R). Evaluation of the %R is based on statistical evaluation of past lab data or guidelines within the methods for organic and inorganic analyses.

Representativeness - Representativeness expresses the degree to which data accurately represents the media and conditions being measured. The representativeness of the data from the sampling site will depend on the sampling procedure. Sample collection is the responsibility of the client. Samples will be homogenized, if required, as part of the laboratory sample preparation. By comparing the quality control data for the samples against other data for similar samples analyzed at the same time, representativeness can be determined for this objective.

Comparability - Comparability conveys the confidence with which one set of data can be compared to another. The analytical results can be compared to other laboratories by using traceable standards and standard methodology and consistent reporting units. The Laboratory Quality Assurance Program documents internal performance, and the interlaboratory studies document performance compared to other laboratories.

Completeness - Completeness is a measure of the quantity of valid data acquired from a measurement process compared to the amount that was expected to be acquired under the measurement conditions. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. Additional information will be stored in the laboratories archives, both hard copy and magnetic tape. Quality Assurance Standard Operating Procedures (SOP's) are in place to provide traceabilty of all reported results.

6. Sampling Procedures

In order for meaningful analytical data to be produced, the samples analyzed must be representative of the system from which they are drawn. It is the responsibility of the client to ensure that the samples are collected according to accepted or standard sampling methods.

Specific requirements for the collection, preservation, and handling of Soil Vapor Samples follows:

Preservation and Handling of Soil Vapor Samples

Air samples for the volatile organics will be collected on coconut charcoal sorbant tubes, 100 mg front section + 50 mg back section (SKC part number 226-01). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the sampling flow set at 0.2 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The sealed tubes will be stored under refrigeration conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within seven days of receipt by the laboratory.

Air samples for phenol will be collected on XAD-7 sorbant tubes, 100 mg front section + 50 mg back section (SKC part number 226-30-12-07). The tubes will be supplied by Lancaster Laboratories, Inc. as sealed by the manufacturer. In the field the ends will be cut off; the sampling flow set at 0.1 liters per minute; the volume of air drawn through the tubes (approximately 10 liters); the ends capped with the supplied plastic caps; and identified with the sampling location, time, temperature, and volume flow rate. The sealed tubes will be stored under refrigeration conditions, 4°C, and returned to the laboratory within two days.

In the laboratory, the samples will be kept at 4°C until the analysis is begun. The analysis must be performed within fourteen days of receipt by the laboratory.

7. Sample Custody

A member of our Sample Administration Group will act as sample custodian for the project. To ensure accountability of our results, a unique identification number is assigned to each sample as soon as possible after receipt at the laboratory. When samples requiring preservation by either acid or base are received at the laboratory, the pH will be measured and documented. Samples requiring refrigeration will be stored in our walk-in cooler which is maintained at 4°C. The use of our computer system in tracking samples (by the LLI sample # assignment) will control custody of the sample from receipt until the time of its disposal. The security system on our laboratory building allows us to designate the entire facility as a secure area since all exterior doors are either locked or attended. Therefore, hand-to-hand chain of custody is not part of our routine procedure but, is available upon request. The procedures for sample log-in and chain-of-custody documentation are detailed in the QA Standard Operating Procedures included in Section No. 7 (QA102 and QA104).

QUALITY ASSURANCE OPERATIONS MANUAL
STANDARD OPERATING PROCEDURE
QA-102

Title: Sample Log-in

Purpose:

In order to provide accountability of our results and to prevent sample loss or mix-up, a unique identification number is assigned to each sample.

Scope:

This SOP will cover the procedure used to log-in samples received for analysis.

Procedures:

1. All samples received by laboratory personnel shall be delivered to the Sample Administration Group immediately upon arrival at the laboratory.

2. All client correspondence relating to samples shall also be transferred to the Sample Administration Group. This includes purchase orders, quotes, letters and completed entry request forms.

3. Personnel of the Sample Administration Group shall log the samples into the computer as soon as practical after receipt. The computer will assign a unique identification number to each sample. Samples shall be logged in on the same day they are received with the following exceptions:

   a. Samples received during a holiday or between 6 p.m. on Friday and 6 p.m. on Sunday. These samples shall be logged-in on the next normal work day.

   b. Samples submitted by clients without any indication of the tests to be performed or with unclear or incomplete information. Every effort shall be made to contact the client on the same day as sample receipt.

   If same day entry is not possible, any special storage requirements (e.g., refrigeration) should be observed.

4. Upon assignment of a sample number, the computer will generate a label which shall be attached to the sample container. The information on the label will include the LLI sample number, the client name, the storage location, a list of analyses requested (by analytical method number), a bottle code indicating container and preservative type, and a unique bar code.

5. Addition of preservatives to unpreserved samples will be the responsiblity of the Sample Administration Group. Preservation should be performed immediately after log-in. A list of preservatives required for routine analyses may be found in the Fee Schedule.

6. All entries in preservation notebooks and on client paperwork shall be made in ink. The error correction procedure given in SOP-QA-109 shall be followed for any changes made in this documentation.

7. After samples are logged-in (or preserved, if required) they shall be stored in the computer-assigned location. If the computer-assigned location is inappropriate for the samples, the location code may be changed by manually overriding the computer.

QA102
SOP QA #1

Prepared by: M Louise Hess Date: 5/21/90

Approved by: Glenn M. Cantilly Date: 4 June 90

Read and understood by: _____ Date: _____

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Initiated Date: 3/87
Revised Date: 9/28/90

QUALITY ASSURANCE OPERATIONS MANUAL
STANDARD OPERATING PROCEDURE
QA-104

Title: Chain-of-Custody Documentation

Purpose:

In order to demonstrate reliability of data which may be used as evidence in a legal case or required by a regulatory agency, an accurate written record tracing the possession of the sample from its receipt at the laboratory to the time of its disposal must be maintained.

Scope:

Procedures for initiating and maintaining chain-of-custody documentation are described in this document.

Definition:

A sample is in custody if it is in any one of the following states:

1. In actual physical possession.
2. In view after being in physical possession.
3. In physical possession and locked up so that no one can tamper with it.
4. In a secured area, restricted to authorized personnel.

Procedures:

1. Chain-of-custody documentation shall be kept upon request of the client or for any samples which are known to be involved in a legal dispute. As with all analytical data, it is extremely important that documentation be filled out completely and accurately with every transfer. If changes to the form need to be made, the error correction procedure given in SOP-QA-109 shall be followed.

2. If requested by the client, the chain-of-custody documentation will begin with the preparation of bottles. A form (see Attachment 1) will be initiated by the person packing the sample bottles for shipment to the client. If the delivery of bottles is via our Transportation Department, the driver shall sign the form when relinquishing the bottles. Drivers must also sign chain-of-custody forms when picking up samples which require such documentation.

3. When samples arrive at the laboratory, a member of the Sample Administration Group will receive them and sign the chain-of-custody form, if one is provided with samples. If the sample was picked up by our Transportation Department, the driver must sign to indicate relinquishing the sample.

4. Samples will be logged into the computer as described in QA-102. Sample Administration personnel shall indicate locked storage, enter a lab note to inform analysts of the need for chain-of-custody documentation, and enter the analysis number for "laboratory chain-of-custody".

5. Sample Administration personnel shall initiate a "Laboratory Chain-of-Custody" form (Attachment 2) for each type of container in the sample, and relinquish the samples to a sample custodian or designated key holder, who will store the sample in the assigned locked location. At this point, external chain-of-custody forms will be filed with the Accounts Receivable Department to be returned with the invoice, and the internal forms will accompany the samples.

6. Sample handling should be kept to a minimum. Analysts requiring use of a sample will requisition it through the computer requisition program. During the hours where sample support is manned by sample custodians, the custodian will receive the computerized requisition, remove the sample from storage and sign the "released by" column to indicate the sample has been relinquished. The analyst shall sign the "received by" column and note the reason for change of custody <u>before</u> taking the samples to their work area. It will be a shared responsibility of technicians and sample custodians to ensure that forms are signed with each transfer.

All changes of custody must be documented on the form. The following changes of custody shall be handled as follows:

a. Signatures involving transfers from one shift to another shall be the responsibility of the technician who originally acquired the sample from sample support. When samples are then returned to storage, the person returning the samples shall be responsible to sign the "released by" column, and to ensure that samples were properly received by the custodian with his/her signature in the "received by" column.

b. Occasionally a sample will be needed for analysis by a technician in a department while it has been signed out to a technician in another department. It will be the responsibility of the first technician who received the sample to see that the second technician needing the sample signs for receipt and return of the sample to them.

c. Weekend work hours do not always have a sample custodian available. During these times the Lancaster Labs security personnel function as key holders to the storage areas. Technicians requiring use of samples over these times <u>must</u> obtain signatures from security personnel, in place of regular sample custodians. It may be necessary to page the security staff on weekends to acquire their signatures and assistance.

d. Some samples are released by sample support and stored temporarily in other areas of the laboratory e.g. GC/MS Volatiles. During this time they may be worked on by several people in that department. Each of these people must sign for change of custody. These samples when completed are then returned to sample support. It will be the responsibility of the department who held temporary storage to see that all necessary signatures are on the chain of custody form <u>before</u> returning samples and forms, at the same time, to sample support. It is also important to return these sample groups as soon as possible after verification of data, because the chains may be required for data packages.

7. Analysts in possession of samples shall remove the aliquot required for analysis and return the sample to storage as described in #8 below with a minimum of delay. During the time of possession, samples must remain in the analyst's view or be locked-up. If additional containers of the sample are created (e.g., an extract container from preparation for organic analysis), an additional form marked with the container type shall be created to accompany the new container.

8. After analysis, samples shall be relinquished to a key holder or sample custodian who will return the samples to locked storage. The forms which remain with the samples shall be signed again to indicate storage, and the sample custodian shall review the forms to ensure that all transfers are completely documented. Sample custodians shall not return a sample to its storage location without signing an accompanying chain.

9. After completion of analysis, these forms are given to the Data Package Group for inclusion in extended reports.

QA104
SOP QA #1

Prepared by: M. Louise Hess Date: 10/2/90

Approved by: [signature] Date: 2 Oct 90

Read and understood by: _____ Date: _____

Lancaster Laboratories INCORPORATED
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Locked Storage Internal Chain of Custody

ORIGINAL SAMPLE

Client/Project: _____

Preservative: _____ Matrix: _____
Analyses:

Sample # Range: _____

Storage Location: _____

Sample Number(s)	Released by	Received by	Date	Time	Reason for Change of Custody

This form has been designed to accompany the sample from the moment it is originally entered into the computer until the last test is verified.

2016 Rev. 12/19/89

Lancaster Laboratories
INCORPORATED
2425 New Holland Pike, Lancaster, PA 17601 • 717/656-2301

Chain of Custody

Lancaster Laboratories Sample Number

Client: P.O. No.:

Work Order No.: Project Name:

Submit Report to: FSC:

Sampler: Project Location: Analyses

Sample Type:	
HZ	Hazardous
SO	Soil
PW	Potable Water
GW	Ground Water
SW	Surface Water
WW	Waste Water
SL	Sludge

Field sample number / sample identification	Date	Time	COMP	GRAB	Number of Containers (Total)	Sample Type (use reference)							Remarks:

Sample Relinquished by:	Date	Time	Sample Received by:	Date	Time	Reason for Transfer

Section No. 7
Page 9 of 9

2102 4/89

Copies: White and Yellow copies accompany samples to Lab. Pink copy retained by Sampler.

8. Calibration Procedures

Procedures for initial calibration and continuing calibration verification are in place for all instruments within the laboratory. The calibrations generally involve checking instrument response to standards for each target compound to be analyzed. The source and accuracy of standards used for this purpose are integral to obtaining the best quality data. The standards are purchased from commercial supply houses either as neat compounds or as solutions with certified concentrations. The accuracy of these purchased standards is checked by comparing to solutions obtained from USEPA, when available. Most solutions and all neat materials require subsequent dilution to an appropriate working range. All dilutions performed are documented and the resulting solution is checked by obtaining the instrument response of the new solution and comparing with the response to the solution currently in use. Any discrepancies between the responses are investigated and resolved before the new solution is used. Each standard is assigned a code which allows traceability to the original components. The standard container is marked with the code, date prepared and the initials of the preparer. Shelf-life for standards are included in the calibration procedures and new standards are prepared before the expiration date.

Each instrument is calibrated with a given frequency using one or more concentrations of the standard solution. As analysis proceeds, the calibration is checked for any change in instrument response. If the calibration check verifies the initial response, the analysis proceeds. If the calibration check indicates that a significant change in instrument response has occurred, then a new calibration is initiated. If necessary, maintenance may be performed prior to the recalibration.

Calibration records are usually kept in the form of raw data with the other instrument print-outs. In cases where no data system is used, calibration data is manually recorded in notebooks. Any maintenance or repair is also recorded in a notebook. The information recorded either in the notebooks or on the instrument print-out includes the date, employee name and/or identification number, and concentration or code number of standard.

The frequency of calibration and calibration verification, number of concentrations used, and acceptance criteria for each of the instruments to be used are listed on Table 8-1.

Table 8-1

	Initial Calibration			Continuing Calibration Verification		
Instrument	Frequency	# of Standard Concentrations	Acceptance Criteria	Frequency	# of Standard Concentrations	Acceptance Criteria
Gas Chromatograph (Volatiles)	Each Batch	5	RSD $\leq$ 25%	Every 10 Samples	1	% D $\leq$ 15%
HPLC (Phenol)	Each Run	5	Correlation Coefficient $\geq$ 0.995	Every 4 Samples	1	Calib. results will be averaged. Correlation coefficient for linear least squares fit $\geq$0.995

9. Analytical Procedures

The analytical procedures to be used for Volatile Organic Compounds listed in Table 9-1 are those described in NIOSH method 1500 and 1003 (modified). Phenol will be determined according to OSHA method 32. Copies of these methods are included in Section 9.

Volatiles - This method determines the concentration of volatile organic compounds in air. This method involves collection of the vapor sample onto a charcoal tube, desorption with carbon disulfide, and subsequent analysis by gas chromatography.

Phenol - This method is applicable to the analysis of phenol in air. The method involves collection of the vapor sample onto a sorbant tube, desorption with methanol, and subsequent analysis by high performance liquid chromatography (HPLC).

Table 9-1

Estimated limit of quantitation (LOQ) for soil vapor study *

Analyte	Estimated LOQ** ppm (v/v)	Required LOQ*** ppm (v/v)
Acetone	0.35	254
Chlorobenzene	0.29	100 #
Chloroform	0.46	496
1,1-Dichloroethane	0.32	3.4
1,1-Dichloroethylene	0.30	515
Ethyl Benzene	0.13	9316
Methyl Ethyl Ketone	0.30	13
Methyl Isobutyl Ketone	0.22	233
Methylene Chloride	0.63	22.4
Tetrachloroethylene	0.61	16.8
Toluene	0.21	36556
1,1,1-Trichloroethane	0.40	2819
1,1,2-Trichloroethane	0.40	1.1
Trichloroethylene	0.45	71.5
Xylenes (all isomers)	0.20	4794
Phenol	0.20	1.4

* Assumes a 10 liter sample volume and two 100mg/50mg charcoal tubes or one 100 mg/50 mg XAD-7 tube used for sampling.

** Based on 10 times the MDL.

*** Based on the concentrations of soil vapor at equilibrium with acceptable soil concentrations.

No specific concentration is listed for chlorobenzene.

Selected Organic Compounds on Charcoal Tube for ECC Site - Soil Vapor Analysis

References:

NIOSH Methods 1003, 1005, 1015, 1022, 1300, 1500 and P&CAM 127.

Principle:

Organic vapors in soil gas are collected on charcoal tubes by passing the gas through the charcoal tubes at a controlled rate for a specified period of time. The volatile organic compounds are removed from tube by desorbing the tube with carbon disulfide and analyzing the solvent by gas chromatography. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of volatile organic compounds sorbed on charcoal tubes. Two 100 mg/50 mg charcoal tubes will be analyzed for each vapor collection sample. The list of the specific compounds follows. The methods listed above are the NIOSH methods which include this list of analytes. The sampling and analysis conditions are the same for the NIOSH methods, so for the purposes of this analysis, the analytes will all be determined using the same analytical method.

<u>Analytes:</u>
Acetone
Chlorobenzene
Chloroform
1,1-Dichloroethane
1,1-Dichloroethylene (Vinylidene chloride)
Ethyl Benzene
Methyl Ethyl Ketone
Methyl Isobutyl Ketone
Methylene Chloride (Dichloromethane)
Tetrachloroethylene (Perchloroethylene)
Toluene
1,1,1-Trichloroethane (Methyl Chloroform)
1,1,2-Trichloroethane
Trichloroethylene
Xylenes (ortho, meta, and para isomers)

Apparatus and Reagents:

1. Hewlett-Packard 5890 series Gas Chromatograph with FID detection (or equivalent). The instrument must be configured with capillary column capability. Dual auto samplers and detectors may be used to allow for dual column operations. The gas chromatographic system will be equipped with an integrator/data system which allows for calibration of the instrument and quantification of the chromatograms using external standards.

2. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um SPB-5 bonded phase (or equivalent).

3. Fused silica capillary gas chromatographic column: 30 meters long with 0.32mm internal diameter with 1.0um DB-WAX bonded phase (or equivalent).

4. Carbon Disulfide: glass distilled, HPLC/Spectral grade (or equivalent). This material has been shown to contain benzene. A further cleanup of the solvent is required if benzene is determined. The benzene is removed using 13X molecular sieves. Approximately 50 grams of the molecular sieves are added to a 2.5 liter bottle of carbon disulfide and the bottle mixed by swirling. The materials are equilibrated overnight. The molecular sieves are removed by filtering the carbon disulfide. The molecular sieves are discarded after allowing the carbon disulfide to evaporate in a hood. The carbon disulfide is returned to the original bottle and a second quantity of molecular sieves added. This process is repeated a total of five times. The benzene concentration must be below 1 ug/mL in the cleaned up solvent.

5. Reagent grade standards for all analytes listed above. The neat materials must have a listed purity of greater than 95%.

6. Desorption vials: 4 mL vials with screw top lids with PTFE or other material which is impervious to carbon disulfide.

7. Autosampler vials: 1.5 mL vials to fit autosampler on gas chromatograph. Either a screw top or crimp closure can be used. The septum must be PTFE lined and must not contribute any components to the solvent blank used for the analysis.

Chromatographic Conditions:

Injector temperature:	250 C
Detector temperature:	320 C
Detector Range :	2^5
Detector Attenuation:	2^-2
Flow Rate (approximate):	2 mL/min
Purge flow:	3 mL/min
Splitter flow:	90 mL/min
Temperature Program:	
Initial	35 C - 4 min.
Program rate	9 C/min
Final	200 C - 1 min
Inlet Pressure:	3.5 psi
Injection volume:	2 uL (split)

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, at minimum once a week. This will include inspection and replacement of the septum, injector syringe, and other consumable items. This routine maintenance will be recorded in the instrument log book. Clean up, replacement of columns and other non-routine maintenance will also be recorded in the instrument log.

Safety Precautions:

Carbon disulfide is extremely flammable and considered toxic. The OSHA TLV (an eight hour TWA) is 10 ppm. Both inhalation and skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Many of the analytes also are inhalation hazards. 1,1-Dichloroethylene, 1,1,2-trichloroethane, trichloroethylene, chloroform, and tetrachloroethylene have been tentatively classified as known or suspected human or mammalian carcinogens. The handling of the neat materials should be performed with gloves and be in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.1 g for nonchlorinated compounds and 0.3 g for chlorinated compounds into a 10 mL volumetric containing approximately 2 mL of carbon disulfide. The standards are prepared in the order listed below. This order is roughly the reverse of the vapor pressure to prevent excessive evaporation of the more volatile compounds. After all the compounds are weighed into the volumetric, dilute to 10 mL with carbon disulfide.

Chlorobenzene
Tetrachloroethylene
Xylenes
Ethyl Benzene
Trichloroethylene
Toluene
1,1,2-Trichloroethane
1,1,1-Trichloroethane
Methyl Isobutyl Ketone
Methyl Ethyl Ketone
Chloroform
Acetone
Methylene Chloride
1,1-Dichloroethane
1,1-Dichloroethylene

Working calibration standards are prepared at the following dilutions in carbon disulfide:

DF 20	0.5 mL stock diluted to 10 mL	500 mg/l
DF 40	0.25 mL stock diluted to 10 mL	250
DF 100	0.1 mL stock diluted to 10 mL	100
DF 400	0.5 mL of DF 20 solution diluted to 10 mL	25
DF 2000	0.25 mL of DF 20 solution diluted to 25 mL	5
DF 4000	1.0 ml of DF 400 solution diluted to 10 mL	2.5

All measurement of the stock and working solutions' volume prior to dilution should be using pipets or syringes. After all the analytes have been added, add carbon disulfide to make the total volume 10 mL. After the stock and working standards are prepared they are transferred to a glass vial with a PTFE lined lid and stored in a freezer. The stock standards can be stored for up to 30 days under freezer conditions, the working standards can be stored for up to 1 week in the freezer before being replaced.

Preparation of a surrogate standard:

Weigh 0.15 g of n-butanol into a 10 mL volumetric containing 2 mL of carbon disulfide. After the neat surrogate material is weighed into the volumetric, dilute to 10 mL with carbon disulfide. Transfer the solution to a vial with screw cap and PTFE lined lid. Stored in a freezer the solution is stable for 1 week.

Preparation of a spiking standard:

Prepare a stock spiking solution as described above for the stock analyte solution. This will be a separate solution than the stock calibration standard. The same storage conditions and times apply as for the calibration standard.

Procedure:

1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within seven days from receipt.

2. Intact charcoal tubes are scored with a file and broken open at each end. The glass wool plug is removed with a fine wire hook. The charcoal from each section of the first tube is combined, added to a 4 mL vial and identified as the "front". Similarly, the charcoal from the second tube in line is combined, added to a 4 mL vial and identified as the "back".

3. To each vial containing charcoal, carbon disulfide is accurately transferred with a syringe. For each 100 mg/50 mg tube analyzed as one section, use three mL of carbon disulfide for the desorption. Add the surrogate standard solution at this point, 20 microliters for the 3.0 mL volume of carbon disulfide. Immediately cap the vial and shake the vial for at least 30 seconds. Desorption should be complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.

4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a GC autosampler vial. Be sure to identify the "front " and "back" sections on the GC autosampler vials. Cap the vials immediately.

5. Prepare the working calibration standards in GC autoinjector vials, adding approximately 1.0 mL of the working solutions to a vial. Five levels of standards are used (DF 20, DF 40, DF 100, DF 400, DF 2000). A DF 4000 standard is used to determine the quantification limit. A complete set of standards are analyzed each sample batch, and a check standard made up of the mid level (DF 100) standard is analyzed at least once for every ten samples.

6. Prepare two spiked tubes as described below:

A. Score intact charcoal tubes with a file and break open at each end. Remove the glass wool plug with a fine wire hook. The charcoal from each section of a single tube is removed as described above, combined, and added to a 4 mL vial.

B. To each vial containing charcoal transfer carbon disulfide with a syringe measuring the volume exactly. Three mL of carbon disulfide is used for desorption of all the carbon in the 100 mg/50 mg tube. Add the surrogate standard solution at this point, 20 microliters to the 3.0 mL volume of carbon disulfide. Add the spiking standard solution, 10 uL to the 3.0 mL volume of carbon disulfide.

Immediately cap the vial and shake the vial for at least 30 seconds. Desorption is complete after 30 to 45 minutes. The vial should be mixed by shaking at least two times during this period. Allow the charcoal to settle to the bottom of the vial before removing the solvent.

C. Transfer between 1.0 and 1.5 mL of the solvent from each vial used for desorption to a GC autosampler vial. Cap the vials immediately.

7. Prepare a media blank by desorbing a sealed charcoal tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.

8. Prepare a solvent blank by adding 1.5 mL of the carbon disulfide plus 10 uL of the surrogate standard to a GC autosampler vial. Cap the vial immediately.

9. Analyze the calibration standards, a solvent blank (with surrogate standard added), the DF 4000 quantification limit standard, the check standard (roughly one every 10th sample) and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples). A MDL study is included in Table 3. The expected detection limit is listed in Table 1.

10. After the samples have been analyzed, review the retention times for all calibration and check standards. If the retention times are within 0.04 minutes or 0.2% of retention time (whichever is larger), the initial retention times can be used to identify the components. If the retention times vary more than this amount the retention times should be updated based on each check standard.

11. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.

12. Calibrate the system based on the peak height of the five levels of standards. Calculate an average response factor based on the amount in the standards (in ug/mL) per peak height unit. The average response factor is used to calculate the concentration as long as the response factor varies less than 25% from the average. If the variation is greater than 25%, a calculation based on the response factor of the standard which is closest to the peak height of the analyte is required.

13. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the following formula:

mg analyte/tube = [(A)xCFxDxDV/1000]

where A = sample peak height
CF = calibration factor {amount (ug/mL)/peak height}
D = dilution factor
DV = desorption volume in mL

The quantity of analyte in the vapor phase is calculated using the following formula:

ppm (v/v) analyte = [(M/V)x(24.45/MW)x((T+273)/298)x(760/P)]

where M = mg analyte/tube
V = volume collected in cubic meters
T = temperature in C
P = pressure in mm Hg
MW = molecular weight of the analyte

Quality Control:

Spikes: Two unopened tubes will be spiked with the compounds of interest and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) must be less than 15%. The spiking levels are approximately 0.1 and 0.3 mg per tube. No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to the DF 4000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

Surrogate standard: A surrogate standard, n-butanol, will be added to each sample, spike and blank at approximately 0.1 mg/mL of desorption solvent. The surrogate recovery should be within 25% of the expected value. If problems with the surrogate standards are noted but the spikes of tubes give acceptable results, an alternate surrogate material may be used.

Check standard: The mid level (DF100) standard will be used as a check standard. This will be analyzed after every tenth sample, at least one check standard will be analyzed per batch. For a batch of 11 to 20 samples, two check standards will be analyzed. This standard must be within 15 % of the expected value. If it is not, repeat the analysis of the check standard or a fresh supply of the standard. If that is within the acceptance range report the new standard. If that is still outside the acceptance range, repeat the calibration standards and all samples since the last check standard which was within the acceptance range. If it is not possible to repeat the analysis the data must be flagged with a comment on the report.

Quality Control Samples used to assess the analyses:

Media blanks	(MB)
Solvent blanks	(SB)
Field blanks	(FB)
Travel blanks	(TB)
Spiked tube	(MS)
Duplicate spiked tube	(MSD)

The solvent blank is used to determine the possible contamination of samples while in the laboratory. The solvent blank is generated in the laboratory.

The field blank and travel blank are used to determine the possible contamination of samples while in the field. These blanks are generated in the field.

The media blank is used to correct the spiked tube and duplicate spiked tube for any contaminants found in the media. The media blank is generated in the laboratory from the same batch of media as was used in the field.

The spiked tube and duplicate spiked tube are used to assess the precision and accuracy of the analysis. The spiked tube and duplicate spiked tube are generated in the laboratory.

Table 1

Limit of quantitation for soil vapor analyses

In air calculation: Selected VOCs on Charcoal tubes
Limit of Quantitation (LOQ)

FLOWRATE: .2 (L/min) Atm P: 760
TIME: 50 (min)
VOLUME: 10 (Liters)
TEMP (C): 25 Desorb Vol: 3 mL

Compound	LOQ ppm	LOQ mg/m3	Analyte LOQ (mg/tube)
Acetone	.35	.82	.0082
Chlorobenzene	.29	1.36	.0136
Chloroform	.46	2.24	.0224
1,1-Dichloroethane	.32	1.28	.0128
1,1-Dichloroethene	.30	1.18	.0118
Ethyl benzene	.13	.56	.0056
Methyl ethyl ketone	.30	.88	.0088
Methyl isobutyl ketone	.22	.90	.0090
Methylene chloride	.63	2.20	.0220
Tetrachloroethylene	.61	4.16	.0416
Toluene	.21	.78	.0078
1,1,1-Trichloroethane	.40	2.20	.0220
1,1,2-Trichloroethane	.40	2.16	.0216
Trichloroethylene	.45	2.42	.0242
Xylenes	.20	.88	.0088

LOQ is based on an MDL study for the analysis.

In air calculation: Phenol on XAD-7 tube
Limit of Quantitation (LOQ) calculation

FLOWRATE: .1 (L/min) Atm P: 760
TIME: 100 (min)
VOLUME: 10 (Liters)
TEMP (C): 25 Desorb Vol: 3 mL

Compound	LOQ ppm	LOQ mg/m3	Analyte LOQ (mg/tube)
Phenol	.20	.76	.0076

LOQ is based on an MDL study for the analysis.

Table 2

Dynamic Range: Selected VOCs on charcoal tubes

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit ppm	Upper Limit ppm
Acetone	.015	1.50	.632	63.2
Chlorobenzene	.045	4.50	.974	97.4
Chloroform	.045	4.50	.925	92.5
1,1-Dichloroethane	.045	4.50	1.112	111.2
1,1-Dichloroethene	.045	4.50	1.135	113.5
Ethyl benzene	.015	1.50	.346	34.6
Methyl ethyl ketone	.015	1.50	.509	50.9
Methyl isobutyl ketone	.015	1.50	.367	36.7
Methylene chloride	.045	4.50	1.294	129.4
Tetrachloroethylene	.045	4.50	.663	66.3
Toluene	.015	1.50	.399	39.9
1,1,1-Trichloroethane	.045	4.50	.827	82.7
1,1,2-Trichloroethane	.045	4.50	.827	82.7
Trichloroethylene	.045	4.50	.840	84.0
Xylenes	.015	1.50	.346	34.6

If levels greater than the upper limit are found on the charcoal tube, the extract will be diluted to bring the concentration within the dynamic range of the analysis.

Dynamic Range: Phenol on XAD-7 tube

Compound	Lower Limit mg/tube	Upper Limit mg/tube	Lower Limit ppm	Upper Limit ppm
Phenol	.008	.76	.197	19.7

If levels greater than the upper limit for the analysis are detected, the tube extract will be diluted to bring the concentration within the analytical dynamic range.

Table 3

MDL calculation: Selected Organics

Analyte	Std. Conc. (ug/mL)	# Reps	SD (n-1)	MDL (ug/tube)	PQL (ug/tube)
Acetone	5.08	7	0.0742	0.413	4.1
1,1-Dichloroethylene	10.06	7	0.1059	0.589	5.9
1,1-Dichloroethane	10.14	7	0.1146	0.638	6.4
Methyl Ethyl Ketone	5.17	7	0.0791	0.440	4.4
Chloroform	10.33	7	0.2007	1.117	11.2
1,1,1-Trichloroethane	10.03	7	0.1993	1.109	11.1
Carbon Tetrachloride*	10.33	7	0.2480	1.380	13.8
Trichloroethylene	10.03	7	0.3080	1.714	17.1
Methyl Isobutyl Ketone	5.31	7	0.0815	0.453	4.5
Toluene	5.38	7	0.0704	0.392	3.9
1,1,2-Trichloroethane	10.29	7	0.1940	1.080	10.8
Tetrachloroethylene	10.22	7	0.3746	2.084	20.8
Chlorobenzene	10.20	7	0.1227	0.683	6.8
Ethylbenzene	5.38	7	0.0501	0.279	2.8
p-Xylene	1.74	7	0.0573	0.319	3.2
m-Xylene	1.70	7	0.0484	0.269	2.7
o-Xylene	1.86	7	0.0782	0.435	4.4

* Carbon Tetrachloride studied rather than methylene chloride. The MDL for these two compounds is similar.

Analysis of Phenol on XAD-7 Tube for ECC Site - Soil Vapor Analysis

References:

OSHA Method 32

Principle:

Organic vapors in soil gas are collected on XAD-7 tubes by passing the gas through the tubes at a controlled rate for a specified period of time. Phenol is removed from tube by desorbing the tube with methanol and analyzing the solvent by high performance liquid chromatography using a ultraviolet (UV) detector at 218 nm. Knowing the exact volume of soil gas passed through the tube and the mass of the organic compound sorbed on the tube, the concentration of the compound in the soil gas can be calculated.

Scope:

This method is applicable to the analysis of phenol sorbed on XAD-7. One 100 mg/50 mg XAD-7 tube will be analyzed for each vapor collection sample.

Apparatus and Reagents:

1. A Shimadzu high performance liquid chromatograph (HPLC) equipped with a sample injector, reverse-phase HPLC column (see item 2), variable wavelength detector, integrator/data system. The data system allows for calibration of the instrument and quantification of the chromatograms using external standards.

2. Reverse phase stainless steel column (25 cm long X 4.6 mm ID), HPLC column packed with Whatman 5 ODS 3 packing material (or equivalent).

3. HPLC grade methanol.

4. Deionized water.

5. Reagent grade phosphoric acid (H_3PO_4).

6. Reagent grade standard of phenol from Chem Service or other supplier which indicates the purity.

7. Glass sampling tubes of approximately 4 to 5 cm in length (4mm ID x 6mm OD) which are packed with 100-mg front section, and a 50-mg back section of 15/50 mesh XAD-7 resin. Small silanized glass wool plugs are placed in the ends and in the middle between the sections of resin. SKC part number 226-30-12-07.

Chromatographic Conditions:

Injector volume:	25 uL
Detector wavelength:	218 nm
Mobile Phase:	59/41 (v/v) methanol/water mixture with 0.1% H3PO4.
Flow Rate:	1 mL/min.

Instrument Maintenance:

Routine instrument maintenance will be carried out on a regular basis, as described in Section 13.0 of this Appendix.

Safety Precautions:

Methanol is flammable and must be handled with care. Skin exposure should be avoided. Large quantities of the solvent should be handled in a hood.

Preparation of calibration standards:

Stock calibration standards: Weigh approximately 0.12 g of phenol into a 25 mL volumetric and dilute to volume with methanol. Working calibration standards are prepared at the following dilutions in methanol:

DF 200	1.0 mL stock diluted to 200 mL	(24 ug/l
DF 100	1.0 mL stock diluted to 100 mL	(48 ug/l)
DF 50	1.0 mL stock diluted to 50 mL	(96 ug/l)
DF 25	1.0 mL stock diluted to 25 mL	(192 ug/l)
DF 12.5	2.0 mL stock diluted to 25 mL	(384 ug/l)

A quantification limit standard (DF 10000) will be prepared by diluting 1 mL of the DF 100 standard into 100 mL with methanol. All measurement of the of the stock and working solutions' volume prior to dilution should be using pipettes. Transfer the standard solutions in brown bottles with Teflon-lined caps for storage in the refrigerator for 15 days.

Calibration:

The standard curve will be injected at a minimum in duplicate throughout the analytical run. A standard will be injected at least after every fourth sample injection. A least squares line will be determined from the standard curve to calibrate the integrator. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration. The percent difference in response between two standard injections of the same standard concentration should be $\leq$ 10%. The RSD between three or more standard injections of the same standard concentration should be $\leq$ 5%.

Preparation of a spiking standard:

Prepare a stock spiking solution as described above for the stock analyte solution. This will be a separate solution than the stock calibration standard. Prepare a working spike standard

by diluting 1 mL of the stock spike standard to 100 mL with methanol (DF 100). The final spiking standard is prepared by diluting the DF 100 solution 1 mL to 100 mL of methanol (DF 10000). The same storage conditions and times apply as for the calibration standard.

Procedure:

1. Samples upon receipt are placed into refrigerated storage until analysis. Analysis will occur within fifteen days from receipt.

2. Transfer the front glass wool and sorbent section of the sampling tube to a 4-mL vial. Label as front section.

   Place the remaining backup section including both glass wool plugs into a separate 4-mL vial. Label as back section.

3. Add 2 mL of methanol to each vial. Immediately cap the vial, and shake it on a mechanical shaker for 15 minutes.

4. Transfer between 1.0 and 1.5 mL of the desorption solvent from each vial used for desorption to a HPLC autosampler vial. Be sure to identify the "front " and "back" sections on the HPLC autosampler vials. Cap the vials immediately.

5. Transfer the working calibration standards in HPLC autosampler vials. Five levels of standards are used. Two vials of each standard (at a minimum) are injected with each batch. A standard is injected at least after every fourth sample injection.

6. Prepare two spiked tubes as described below:

   A. Use the DF 10000 spiking standard solution to prepare the spiked samples.

   B. To two clean XAD-7 sampling tubes, add 1.6 microliters of the spiking solution to each tube. Desorb the spiked samples starting with step 2 of the analytical procedure.

7. Prepare a media blank by desorbing a XAD-7 sampling tube which is from the same lot as the tubes used for the matrix spike/matrix spike duplicate analysis. The procedure is the same as that identified in sections 2-4. This result will be used to calculate the recovery of analyte from the spiked tubes.

8. Prepare a solvent blank by adding 1.5 mL of the methanol to an HPLC autosampler vial. Cap the vial immediately.

9. Analyze the calibration standards, a solvent blank, the DF 10000 quantification limit standard, and the spiked tubes as described above along with the samples for each batch of samples (up to 20 samples).
10. Review the chromatograms, any samples which have analyte responses greater than the highest level standard must be

diluted and reanalyzed. These should be analyzed in the same analytical batch if possible.

11. Calibrate the system based on the peak responses of the five levels of standards. A least squares line will be determined from the standard curve to calibrate the integrator. The correlation coefficient must be greater than or equal to 0.995. The samples will be calculated with the new calibration to determine the phenol concentration.

12. Calculate the recovery from the spiked tubes. The recovery must be within 25% of the expected value. The relative percent difference between the two results for the spiked tubes must also be less than 15%.

Calculations:

The quantity of analyte per tube is calculated using the least squares fit of the Shimadzu integrator. The mg/tube result of the sample is calculated by the integrator following the linear equation of:

$$y = Ax + B$$

(The volume used for desorbing the tube is entered into integrator by the use of dilution factor.)

The quantity of analyte in the vapor phase is calculated using the following formula:

$$\text{ppm (v/v) analyte} = \frac{[(M/V) \times 22.45 \times ((T+273)/298) \times (760/P)]}{94.11}$$

where M = mg analyte/tube
V = volume collected in cubic meters
T = temperature in C
P = pressure in mm Hg
94.11 = MW of Phenol

Quality Control:

Spikes: Two unopened tubes will be spiked with the compound of interest and analyzed with each analytical batch of 20 samples. The recovery must be within 25% of the expected and the relative percent difference (RPD) must be less than 15%. The spiking level is approximately 0.76 ug per tube. No data base of recovery information is available to evaluate the recovery or RPD measurements. Replicate analysis of spiked tubes may be performed to check for analyst error if the spikes can be performed in the same analytical batch. If the recovery or RPD values on the repeat spikes are outside these windows the data will be flagged with a comment on the analytical report.

Blanks: At least one solvent blank will be analyzed per batch of samples. No analyte of interest may be present in the solvent blank at a level equal to the DF 10000 standard. If analytes are found above this level the data will be flagged with a comment on the analytical report.

10. Data Reduction, Validation and Reporting

Raw analytical data generated in the laboratories is collected on printouts from the instruments and associated data system or manually in bound notebooks. Analysts review data as it is generated to determine that the instruments are performing within specifications. This review includes calibration checks, surrogate recoveries, blank checks, retention time reproducibility, and other QC checks described in Section No. 11. If any problems are noted during the analytical run, corrective action is taken and documented.

Each analytical run is reviewed by a chemist for completeness prior to interpretation and data reduction. The following calculations are used to reduce raw data to reportable results.

Volatile Organics in Air - The quantity of analyte per tube is calculated using the following formula:

$$\text{mg analyte/tube} = [(A) \times CF \times D \times DV/1000]$$

Where A = sample peak height
CF = calibration factor [amount (ug/ml)/peak height]
D = dilution factor
DV = desorption volume in ml

The quantity of analyte in the vapor phase is calculated using the following formula:

$$\text{ppm (v/v) analyte} = [(M/V) \times (24.45/MW) \times (T+273)/298) \times (760/P)]$$

Where M = mg analyte/tube
V = volume collected in cubic meters
T = temperature in C
P = pressure in mm Hg
MW = molecular weight of the analyte

Phenol in Air - Include in the calculations the concentration of the analyte found on the front and backup sections of a sampling tube. Express results in mg/m^3 using the following equations:

ug/ml (total) = ug/ml (front section) + ug/ml (backup section)

mg/m^3 = (ug/ml(total)) (2 ml desorption) / air volume in liters

To convert to ppm at 760 mm and 25°C:

$$ppm = (mg/m^3)(24.45)/(MW \text{ of analyte})$$

24.45 is the molar volume of an ideal gas at 760 mm Hg, 25°C.

The principle criteria used to validate data will be the acceptance criteria described in Section No. 11. Following interpretation and data reduction by an analyst, data is transferred to the laboratory sample management system either by direct data upload from the analytical data system or manually. The data is reviewed by the Group Leader or another analyst and verified on the sample management system. The person performing the verification step reviews all data including quality control information prior to verifying the data. If data package deliverables have been requested, the laboratory will complete the appropriate forms (see Appendix A) summarizing the quality control information, and transfer copies of all raw data (instrument print-outs, spectra, chromatograms, laboratory notebooks, etc.) to the Data Packages Group. This group will combine the information from the various analytical groups and the analytical reports from the laboratory sample management system into

one package. This package is reviewed by the Quality Assurance Department for conformance with SOP's and to ensure that all QC goals have been met. Any analytical problems are discussed in the case narrative, which is also included with the data package deliverables.

The validation of the data by the Quality Assurance Department includes spot checking raw data versus the final report, checking that all pertinent raw data is included and does refer to the samples analyzed, review of all QC results for conformance with the method, and review of the case narrative for description of any unusual occurrences during analysis. This validation is performed using techniques similar to those used by the Sample Management Office for the USEPA's Contract Laboratory Program. The validation performed by the laboratory does not address useability of the data, which usually requires some knowledge of the site. The laboratory will make every attempt to meet the requirements of this QAPP, thus reducing the need to assess useability of the data.

The laboratory sample management system is programmed to accept and track the results of quality control samples including blanks, surrogates, recoveries, duplicates, controls, and reference materials. The computer is programmed with the acceptance criteria for each type of QC sample and will display an out-of-spec message if the data is not within specifications. All data outside of specifications appears on a report to the Quality Assurance Department on the next working day. These are reviewed by the Quality Assurance Department for severity of the problems and trends in the data. The reports are then sent to the analytical groups for the purpose of

documenting the corrective action taken. The sample management system also produces control charts and has searching capabilities to aid in data review. The flow of data from the time the samples enter the laboratory until the data is reported are summarized in Table 10-1. Any data recorded manually will be collected in bound notebooks. All entries will be in ink, with no erasures or white-out being permitted. Any changes in data will be made using a single line to avoid obliteration of the original entry and will be dated and signed. Any data resulting from instrument printouts will be dated and will contain the signature and/or identification of the analyst responsible for its generation. After copies of the data are incorporated into the data package deliverables, the originals will be stored in locked archives at the laboratory for a period of ten years.

Table 10-1

SAMPLE AND DATA ROUTING AT LANCASTER LABORATORIES, INC.

Action	Personnel Involved
Sample received at LLI	Sample Administration
Sample is entered onto sample management system (lab ID number assigned, analyses scheduled, chain-of-custody started, storage location assigned)	Sample Administration
Sample stored in assigned location (refrigerator, freezer, etc.)	Sample Administration
Acknowledgement sent to client	Sample Administration
Removed from storage for analysis; tube is desorbed; extracts retained in the laboratory	Technical Personnel
Analysis is performed according to selected analytical method; raw data recorded in notebook and transferred to computer by chemist or technician*	Technical Personnel
Computer performs calculations as programmed according to methods	Data Processing
Chemist or supervisor verifies raw data	Technical Personnel
Data package deliverables are assembled	Data Package Group
Data packages are reviewed prior to mailing	Quality Assurance Dept. Laboratory Management

*Analyses requiring the chemist's interpretation may involve manual data reduction prior to entry onto the computer.

11. Internal Quality Control Checks

The particular types and frequencies of quality control checks analyzed with each sample are defined in the methods in Section 9. The quality control checks routinely performed during sample analysis include surrogates, matrix spikes, and blanks.

Surrogates (used for organic analysis only) - Each sample, matrix spike, matrix spike duplicate, and blank are spiked with a surrogate compound during desorption in order to monitor desorption and analysis. Surrogates are used to evaluate analytical efficiency by measuring recovery.

Matrix Spikes - A matrix (blank sorbant tube) is spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Duplicates (matrix spike duplicate) - A second blank sorbant tube is analyzed at the same time as the original sample in order to determine the precision of the method. Recovery of the original compared to the duplicate is expressed as relative percent differences (RPD).

Blanks (Media, Method) - Blanks are unopened sorbant tubes from the same batch as those used in the field. They are opened in the laboratory and treated with the same reagents and surrogate standards as samples and carried through the entire analytical procedure.

The charts that follow show the types and frequency of QC performed, along with the acceptance limits.

QC Charts

Type	Acceptance Limits	Frequency
VOLATILES BY GC:		
Surrogate:		
n-butanol	85-125	Each sample, MS, MSD, and blank.
Matrix Spike:		
Acetone	75-125	Each group (≤20) of samples.
Chlorobenzene	75-125	
Chloroform	75-125	
1,1-Dichloroethane	75-125	
1,1-Dichloroethene	75-125	
Ethyl benzene	75-125	
Methyl ethyl ketone	75-125	
Methyl isobutyl ketone	75-125	
Methylene chloride	75-125	
Tetrachloroethylene	75-125	
Toluene	75-125	
1,1,1-Trichloroethane	75-125	
1,1,2-Trichloroethane	75-125	
Trichloroethylene	75-125	
Xylenes (sum of isomers)	75-125	
Matrix Spike Duplicate (RPD):		
Acetone	15	Each group (≤20) of samples.
Chlorobenzene	15	
Chloroform	15	
1,1-Dichloroethane	15	
1,1-Dichloroethene	15	
Ethyl benzene	15	
Methyl ethyl ketone	15	
Methyl isobutyl ketone	15	
Methylene chloride	15	
Tetrachloroethylene	15	
Toluene	15	
1,1,1-Trichloroethane	15	
1,1,2-Trichloroethane	15	
Trichloroethylene	15	
Xylenes (sum of isomers)	15	
Blank	≤ level of DF4000 std	Each group (≤20) of samples.

QC Charts
(Continued)

Type	Acceptance Limits	Frequency
PHENOLS BY HPLC:		
Matrix Spike:		
Phenol	75-125	Each group (≤20) of samples.
Matrix Spike Duplicate (RPD):		
Phenol	15	Each group (≤20) of samples.
Blank	≤LOQ	Each group (≤20) of samples.

12. Performance and System Audits

System audits are conducted on each department at Lancaster Laboratories, Inc. (LLI) by members of the Quality Assurance Department. The audits include checks on methodology, reagent preparation, equipment calibration and maintenance, quality control results, and training of personnel. The results of the audits and corrective action, where necessary are communicated to laboratory personnel and management by means of a written report. Audits by outside organizations including clients, regulatory personnel and the USEPA are permitted by arrangement with the Quality Assurance Department.

On a monthly basis, the Quality Assurance Department reviews summaries of the quality control data entered onto the computerized sample management system by analysts. Control charts and statistics are reviewed for trends which may indicate problems with the analytical data. In this way, small problems are identified before they have any significant impact on laboratory results.

Performance audits consist of both intralaboratory and interlaboratory check samples. Blind samples containing known amounts of target analytes are prepared by the Quality Assurance Department and submitted to the laboratories under fictitious client names. In addition, QC samples from EMSL-Cinncinnati are analyzed quarterly to assess laboratory accuracy. LLI also participates in a number of interlaboratory performance evaluation studies which involve analysis of samples with concentrations of analytes that are known to the sponsoring organization, but unknown to the laboratory. Inorganics,

pesticide/herbicides, trihalomethanes, volatile organic compounds, semivolatile organic compounds and traditional wet chemistry analyses are analyzed by LLI for studies conducted by the USEPA and the New York Department of Health. LLI is a contractor to the USEPA under the Contract Laboratory Program which provides laboratory analysis in support of the Superfund program. Part of maintaining this contract includes analysis of quarterly blind samples. Interlaboratory check samples are also provided through the American Industrial Hygiene Association accreditation program. The Proficiency Analytical Testing Program (PAT Program) submits samples to the laboratory quarterly. Charcoal tubes with quantities of organics known to the sponsoring organization are trapped on charcoal tubes at levels unknown to the laboratory. Representative results from some of these PAT Program rounds are attached to this section. Representative results from some of these studies are attached to this section.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
ALUMINUM	1	1420	1428	1150- 1680	1220- 1620	ACCEPTABLE
	2	62.2	45.7	20.6- 85.7	28.8- 77.4	ACCEPTABLE
ARSENIC	1	318	312	252- 373	267- 358	ACCEPTABLE
	2	50.1	51.9	39.4- 61.9	42.3- 59.1	ACCEPTABLE
BERYLLIUM	1	810	806	660- 917	693- 884	ACCEPTABLE
	2	20.1	20.9	16.2- 25.5	17.4- 24.4	ACCEPTABLE
CADMIUM	1	6.10	6.35	4.20- 8.85	4.78- 8.27	ACCEPTABLE
	2	71.0	72.0	60.4- 82.5	63.2- 79.7	ACCEPTABLE
COBALT	1	435	452	382- 509	398- 493	ACCEPTABLE
	2	27.3	27.2	22.6- 32.2	23.8- 30.9	ACCEPTABLE
CHROMIUM	1	10.8	11.0	5.67- 16.3	7.00- 14.9	ACCEPTABLE
	2	101	106	82.0- 124	87.3- 119	ACCEPTABLE
COPPER	1	701	720	638- 739	657- 770	ACCEPTABLE
	2	26.2	25.2	19.9- 31.0	21.3- 29.6	ACCEPTABLE
IRON	1	37.6	32.5	22.5- 48.9	25.8- 45.6	ACCEPTABLE
	2	1210	1230	1070- 1390	1110- 1350	ACCEPTABLE
MERCURY	1	4.76	5.01	4.27- 5.54	4.55- 5.25	ACCEPTABLE
	2	37.2	44.0	33.2- 53.4	35.7- 50.9	ACCEPTABLE
MANGANESE	1	551	551	468- 628	488- 608	ACCEPTABLE
	2	18.6	19.3	15.4- 23.5	16.4- 22.6	ACCEPTABLE
NICKEL	1	932	940	820- 1050	849- 1020	ACCEPTABLE
	2	41.4	41.8	33.8- 49.5	35.8- 47.5	ACCEPTABLE
LEAD	1	31.8	32.2	24.7- 40.7	26.7- 38.7	ACCEPTABLE
	2	1290	1344	1140- 1550	1190- 1500	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
TRACE METALS IN MICROGRAMS PER LITER:						
SELENIUM	1	27.8	30.0	20.1- 36.8	22.2- 34.7	ACCEPTABLE
	2	127	130	90.7- 155	98.9- 147	ACCEPTABLE
VANADIUM	1	57.1	58.1	46.6- 69.8	49.7- 66.7	ACCEPTABLE
	2	246	255	218- 291	228- 281	ACCEPTABLE
ZINC	1	22.2	25.4	17.6- 36.9	20.1- 34.5	ACCEPTABLE
	2	765	768	671- 853	694- 831	ACCEPTABLE
ANTIMONY	3	22.8	24.0	14.4- 31.6	16.6- 29.4	ACCEPTABLE
	4	148	157	110- 201	122- 189	ACCEPTABLE
SILVER	3	1.20	1.20	0.748- 1.61	0.856- 1.50	ACCEPTABLE
	4	11.6	12.4	9.58- 15.2	10.3- 14.5	ACCEPTABLE
THALLIUM	3	10.1	11.0	6.79- 15.9	7.96- 14.7	ACCEPTABLE
	4	64.5	67.9	51.1- 85.1	55.7- 80.5	ACCEPTABLE
MOLYBDENUM	3	9.53	9.58	4.74- 14.3	6.09- 12.9	ACCEPTABLE
	4	59.3	56.0	33.8- 74.7	39.6- 68.9	ACCEPTABLE
STRONTIUM	3	16.6	16.9	13.5- 21.2	14.5- 20.2	ACCEPTABLE
	4	44.8	45.7	36.1- 57.6	39.0- 54.7	ACCEPTABLE
TITANIUM	3	259	260	216- 297	227- 286	ACCEPTABLE
	4	50.0	50.9	38.5- 62.1	41.7- 58.9	ACCEPTABLE
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
PH-UNITS	3	4.64	4.60	4.52- 4.68	4.54- 4.66	ACCEPTABLE
	4	8.21	8.33	7.96- 8.50	8.03- 8.44	ACCEPTABLE
SPEC. COND.	1	65.5	67.8	57.5- 76.4	59.8- 74.0	ACCEPTABLE
(UMHOS/CM AT 25 C)	2	752	770	676- 838	696- 818	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER 4P025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
MINERALS IN MILLIGRAMS PER LITER: (EXCEPT AS NOTED)						
TDS AT 180 C	1	36.5	32.8	11.0- 58.8	16.9- 52.9	ACCEPTABLE
	2	431	412	294- 542	325- 511	ACCEPTABLE
TOTAL HARDNESS (AS CACO3)	1	11.9	12.0	8.28- 15.6	9.19- 14.6	ACCEPTABLE
	2	190	200	183- 216	187- 212	ACCEPTABLE
CALCIUM	1	3.25	3.00	2.49- 3.58	2.63- 3.44	ACCEPTABLE
	2	54.5	55.4	48.1- 62.1	49.8- 60.3	ACCEPTABLE
MAGNESIUM	1	1.08	1.10	0.929- 1.31	0.976- 1.26	ACCEPTABLE
	2	14.8	15.0	12.9- 17.1	13.4- 16.6	ACCEPTABLE
SODIUM	1	5.44	5.45	4.47- 6.50	4.74- 6.33	ACCEPTABLE
	2	50.6	50.7	44.7- 56.0	46.1- 54.5	ACCEPTABLE
POTASSIUM	1	3.04	3.00	2.41- 3.44	2.54- 3.31	ACCEPTABLE
	2	25.9	26.0	21.6- 30.9	22.8- 29.8	ACCEPTABLE
TOTAL ALKALINITY (AS CACO3)	1	5.67	6.69	2.62- 9.74	3.51- 8.85	ACCEPTABLE
	2	45.4	47.3	39.5- 50.4	40.8- 49.1	ACCEPTABLE
CHLORIDE	1	9.73	8.66	7.19- 11.7	7.75- 11.1	ACCEPTABLE
	2	148	142	131- 154	134- 151	ACCEPTABLE
FLUORIDE	1	.173	0.180	0.110-0.256	0.128-0.238	ACCEPTABLE
	2	.782	0.910	0.772- 1.03	0.804-0.997	CHECK FOR ERROR
SULFATE	1	7.50	8.00	5.00- 10.5	5.70- 9.84	ACCEPTABLE
	2	84.3	90.0	74.7- 102	78.1- 98.6	ACCEPTABLE
NUTRIENTS IN MILLIGRAMS PER LITER:						
AMMONIA-NITROGEN	1	8.84	8.76	6.97- 10.4	7.38- 10.0	ACCEPTABLE
	2	1.64	1.60	1.20- 1.99	1.29- 1.90	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY..

PERFORMANCE EVALUATION REPORT DATE: 12/21/90

WATER POLLUTION STUDY NUMBER WP025

LABORATORY: PA009

ANALYTES	SAMPLE NUMBER	REPORT VALUE	TRUE VALUE*	ACCEPTANCE LIMITS	WARNING LIMITS	PERFORMANCE EVALUATION
NUTRIENTS IN MILLIGRAMS PER LITER:						
NITRATE-NITROGEN	1	3.27	3.20	2.52- 3.85	2.58- 3.69	ACCEPTABLE
	2	.648	0.650	0.473-0.828	0.515-0.786	ACCEPTABLE
ORTHOPHOSPHATE	1	.191	0.190	0.145-0.235	0.155-0.224	ACCEPTABLE
	2	4.73	5.30	4.48- 6.10	4.67- 5.90	ACCEPTABLE
KJELDAHL-NITROGEN	3	< 0.20	7.00	5.01- 8.72	5.45- 8.28	NOT ACCEPTABLE
	4	33.1	26.0	19.5- 32.1	21.0- 30.6	NOT ACCEPTABLE
TOTAL PHOSPHORUS	3	< 0.05	8.20	6.32- 9.62	6.72- 9.23	NOT ACCEPTABLE
	4	.611	0.525	0.449-0.772	0.488-0.733	ACCEPTABLE
DEMANDS IN MILLIGRAMS PER LITER:						
COD	1	115	121	95.0- 138	101- 133	ACCEPTABLE
	2	18.1	13.2	3.16- 29.3	10.7- 25.8	ACCEPTABLE
TOC	1	46.9	48.0	40.8- 55.4	42.7- 53.5	ACCEPTABLE
	2	6.92	7.20	5.72- 8.96	6.14- 8.54	ACCEPTABLE
5-DAY BOD	1	82.8	76.5	45.1- 108	52.9- 100	ACCEPTABLE
	2	19.0	12.6	6.15- 18.9	7.74- 17.4	NOT ACCEPTABLE
PCB'S IN MICROGRAMS PER LITER:						
PCB-AROCLOR 1016/1242	2	5.69	6.50	2.29- 8.85	3.13- 8.01	ACCEPTABLE
PCB-AROCLOR 1260	1	3.75	4.27	1.22- 6.16	1.95- 5.52	ACCEPTABLE

* BASED UPON THEORETICAL CALCULATIONS, OR A REFERENCE VALUE WHEN NECESSARY.

Table 13-1
Preventive Maintenance Schedule

Instrument	Preventive Maintenance	Frequency
GC	Injection Syringe	Weekly
	Septum change	Weekly
	Column maintenance	AN
	Clean detector	AN
HPLC	Reproducability Check	6 months
	Lubricate moving parts	3 months
	Inspect & clean line filter	3 months
	Clean Check valve & pump head	6 months
	Replace plunger seal	6 months
	Replace syringe plunger tip	6 months
	Check lamp intensity	6 months

* AN means as needed. Any of these items may be performed more frequently if response during operation indicates this is necessary.

14. Specific Routine Procedures Used to Assess Data Precision, Accuracy and Completeness

Precision - Precision refers to the reproducibility of a method when it is repeated on a second aliquot of the same sample. The degree of agreement is expressed as the Relative Percent Difference (RPD). The RPD will be calculated according to the following equation:

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

D_1 = First sample value
D_2 = Second sample value (Duplicate)

Duplicates will be run on at least 5% of the samples. Acceptance criteria shall be based on statistical evaluation of past lab data. (See Section No. 11.) All Quality Control sample results are entered into the computer and compared with acceptance limits. In addition, there is a monthly review of values on the computer QC system. Data obtained from quality control samples is entered onto our computer system which charts the data, and calculates a mean and standard deviation on a monthly basis. The Quality Assurance Department then reviews this data for trends which may indicate analytical problems. The control charts are graphical methods for monitoring precision and bias over time.

Accuracy - Accuracy refers to the agreement between the amount of a compound measured by the test method and the amount actually present. Accuracy is usually expressed as a percent Recovery (R). Recoveries will be calculated according to the following equations:

$$\text{Surrogate Recovery} = \frac{Qd}{Qa} \times 100$$

Qd = quantity determined by analysis
Qa = quantity added to sample

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

SSR = Spiked Sample Results
SR = Sample Results
SA = Spike added

$$\text{Laboratory Control Sample Recovery} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Surrogate standards are added to each sample analyzed for organics. Spikes and Laboratory Control Samples will be run on at least 5% of the samples (each batch or SDG, $\leq$ 20 samples). Acceptance criteria for the accuracy recoveries shall be based on statistical evaluation of past lab data. (See Section No. 11.) The computer is programmed to compare the individual values with the acceptance limits and inform the analyst if the results meet specification. If the results are not within the acceptance criteria, corrective action suitable to the situation will be taken. This may include, but is not limited to, checking calculations and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with documentation of any QC problems in the Case Narrative.

Where available, EPA Quality Control materials are run at least quarterly to ensure accuracy of the analytical procedure. Repetitive analysis of a reference material will also yield precision data. Accuracy information determined from reference materials is valuable because variables specific to sample matrix are eliminated.

The QC program is capable of charting data for surrogates, spikes, control materials and reference materials. The Quality Assurance Department reviews these charts for any indication of possible problems (ie shift in the mean and standard deviation).

Completeness - Completeness is the percentage of valid data acquired from a measurement system compared to the amount of valid measurements that were planned to be collected. The objective is analysis of all samples submitted intact, and to ensure that sufficient sample weight/volume is available should the initial analysis not meet acceptance criteria. The laboratory's Sample Management System will assign a unique identification number to the sample which tracks and controls movement of samples from the time of receipt until disposal. All data generated will be recorded referencing the corresponding sample identification number. The completeness of an analysis can be documented by including in the data deliverables sufficient information to allow the data user to assess the quality of the results. This information will include, but is not limited to, summaries of QC data and sample results, chromatograms, spectra, and instrument tune and calibration data. Additional information will be stored in the laboratory's archives, both hard copy and magnetic tape.

15. Corrective Action

Whenever any of the data generated falls outside of the established acceptance criteria outlined for instrument tune and calibration (Section No. 8) and Internal QC (Section No. 11), the cause of this irregularity must be investigated, corrected and documented. The documentation will be used to prevent a recurrence of the problem and to inform management of the situation.

If the results are not within acceptance criteria, the appropriate corrective action will be initiated. This may include, but is not limited to, checking calculation and instrument performance, reanalysis of the associated samples, examining other QC analyzed with the same batch of samples, and qualifying results with a comment stating the observed deviation.

A Standard Operating Procedure is in place which outlines the procedures to be followed when quality control data for an analysis falls outside of previously established acceptance limits. All QC data must be entered onto the computerized QC system promptly after its generation and daily "out-of-spec" data is reported via this system. Any data outside the acceptance criteria will be reviewed by the Quality Assurance Department. Where appropriate, the Quality Assurance Department will place outliers in one of three categories:

A. Marginal Outlier
   Data that are outside the 95% confidence interval but within the 99% confidence interval. This category may also be used for QC samples subject to matrix interferences or sample inhomogeneity.

B. Outlier

Data outside the 99% confidence interval and/or observable trends such as a shift in mean and standard deviation.

C. Extreme Outlier

Such data would indicate the system is out of control and no results should be reported to clients; an example would be more than one reference or control falling outside the 99% confidence interval.

The daily out-of-spec reports are then distributed to Group Leaders or their QC Coordinator who will check all supporting data and document their findings and any corrective action taken. Documentation of QC Data will be filed in the departmental QC notebook. In the case of Outliers or Extreme Outliers the Quality Assurance Department may issue a formal request for investigation and corrective action (see sample form that follows). The Quality Assurance Department is responsible for initiating the corrective actions, insuring that the actions are taken in a timely manner, and that the desired results are produced.

The Quality Assurance Department is also responsible for conducting periodic audits which ensure compliance with laboratory SOP's and assist in identifying and correcting any deficiencies. These audits may entail observation as procedures are carried out or a review of records to demonstrate traceability and compliance with all documented record keeping procedures. Follow-up audits verify that proper corrective action has been taken for the identified discrepancy.

No. _____

INVESTIGATION AND CORRECTIVE ACTION FOR QC OUTLIERS

Part I (to be filled out by QA Director)

1. Date
2. LLI sample number(s) involved
3. Nature of QC outlier
4. _____ Check if investigation must be complete before reporting further data to clients.

Signed _____
Quality Assurance Director

Part II

1. Steps taken to investigate outlier:
2. Explanation of probable cause of outlier:
3. Steps taken to prevent future occurrence:
4. Name of analyst who performed work:
5. Signed _____ Date _____

   Return by _____

16. Quality Assurance Reports to Management

Reports of quality status from the Quality Assurance Department to management are made frequently and in various forms. All results from internal or external performance evaluation samples are circulated to management. A report of each audit performed is prepared and copied to management. Monthly summaries of data obtained from analysis of quality control check samples are generated via the computerized sample management system. These summaries include mean and standard deviation to aid in assessment of data accuracy and precision. Forms summarizing problems which require investigation and corrective action are completed by Group Leaders and circulated to management. Through these channels, laboratory management is kept apprised of QA/QC activities.

Any problems or unusual observations that occur during the analysis of samples for a specific project will be listed on the laboratory report and/or in the case narrative delivered with the data package. The items often discussed in this manner include samples with surrogate recovery outside of the acceptance criteria and samples with matrix problems requiring dilution and causing increased detection limits. Where applicable, any corrective action attempted or performed to address the problem will also be presented.

The laboratory will contact the client for direction regarding major problems such as samples listed on the chain of custody but missing form the shipping container, samples which arrive broken or are accidentally broken in the laboratory, and samples with severe matrix problems. The client will be contacted if it is necessary to change any item in the original project plan.

Appendix A

Example Reporting Forms

Tier I Data Package

Title Page
Table of Contents
Sample Analysis Request Form, Field Chain of Custody
Internal Chain of Custody
Laboratory Chronicle
Method Summary/References
Analytical Reports for Samples and QC Samples
Case Narrative
QC Summary
- GC/MS tuning summary
- Surrogate recovery summary
- Blank results
- Matrix spike/matrix spike duplicate/duplicate results
- LCS results (if applicable)
- Internal standard area summary (GC/MS)

Sample Data
- All raw sample data including instrument printouts (i.e., chromatograms, quant. reports, spectra, etc.)

Standards Data
- Initial calibration summary and supporting raw data
- Continuing calibration summary and supporting raw data
- Standardization data

Raw QC Data
- Raw tune data (GC/MS)
- Blank raw data
- Matrix spike/matrix spike duplicate/duplicate raw data
- LCS raw data (if applicable)

Extraction/Digestion Logs

ANALYSIS REPORT

Lancaster Laboratories INCORPORATED

2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

00649 0

LLL Sample No. A01728128

Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

Date Reported 10/28/91
Date Submitted 10/16/91
Discard Date 10/16/91

Primary Air Sample
50 min. 0.2 L/min 10 L 25C

P.O.
Rel.

ANALYSIS	RESULT AS RECEIVED			LIMIT OF QUANTITATION	LAB CODE
Acetone	0.005	J	mg/tube	0.008	900100000
Chlorobenzene	N.D.		mg/tube	0.01	900200000
Chloroform	N.D.		mg/tube	0.02	900300000
1,1-Dichloroethane	N.D.		mg/tube	0.01	900400000
1,1-Dichloroethylene	N.D.		mg/tube	0.01	900500000
Ethyl Benzene	0.016		mg/tube	0.006	900600000
Methyl Ethyl Ketone	N.D.		mg/tube	0.009	900700000
Methyl Isobutyl Ketone	0.006	J	mg/tube	0.009	900800000
Methylene Chloride	N.D.		mg/tube	0.02	900900000
Tetrachloroethylene	N.D.		mg/tube	0.04	901000000
Toluene	0.024		mg/tube	0.008	901100000
1,1,1-Trichloroethane	N.D.		mg/tube	0.02	901200000
1,1,2-Trichloroethane	N.D.		mg/tube	0.02	901300000
Trichloroethylene	N.D.		mg/tube	0.02	901400000
Xylenes	0.048		mg/tube	0.009	901500000
Acetone in Air	0.2	J	ppm	0.4	901600000
Chlorobenzene in air	N.D.		ppm	0.3	901700000
Chloroform in air	N.D.		ppm	0.5	901800000
1,1-Dichloroethane in air	N.D.		ppm	0.3	901900000
1,1-Dichloroethylene in air	N.D.		ppm	0.3	902000000
Ethyl Benzene in air	0.4		ppm	0.1	902100000
Methyl Ethyl Ketone in air	N.D.		ppm	0.3	902200000
Methyl Isobutyl Ketone in air	0.15	J	ppm	0.2	902300000
Methylene Chloride in air	N.D.		ppm	0.6	902400000
Tetrachloroethylene in air	N.D.		ppm	0.6	902500000
Toluene in air	0.6		ppm	0.4	902600000
1,1,1-Trichloroethane in air	N.D.		ppm	0.4	902700000
1,1,2-Trichloroethane in air	N.D.		ppm	0.4	902800000
Trichloroethylene in air	N.D.		ppm	0.5	902900000
Xylenes in air	1.1		ppm	0.2	903000000

1 COPY TO Kathy Loewen

The American Association for Laboratory Accreditation
Chemical, Biological & Environmental Tests of testing.

Member: American Council of Independent Laboratories, Inc.

Questions? Contact Environmental Client Services at (717) 656-2301
129 00649 0.00 000000

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions

Respectfully Submitted
Lancaster Laboratories, Inc.
Reviewed and Approved by:

Richard C. Entz, Group Leader
Misc. Organics/Ind. Hygiene

ANALYSIS REPORT

Lancaster Laboratories INCORPORATED
2425 New Holland Pike, Lancaster, PA 17601-5994 (717) 656-2301

00649 0

LLI Sample No. A01728129

Lancaster Laboratories, Inc.
2425 New Holland Pike
Lancaster, PA 17601-5994

Date Reported 10/28/91
Date Submitted 10/16/91
Discard Date 10/16/91

#2 Air Sample
100 min @ 0.1 L/min 10 liters 25 C

P.O.
Rel.

ANALYSIS	RESULT AS RECEIVED		LIMIT OF QUANTITATION	LAB CODE
Phenol	N.D.	mg/tube	0.008	900100000
Phenol in Air	N.D.	ppm	0.2	900200000

1 COPY TO Kathy Loewen

The American Association for Laboratory Accreditation
...emical, Biological & Environmental
...ds of testing.

...mber: American Council of
...ndependent Laboratories, Inc.

Questions? Contact Environmental Client Services at (717) 656-2301
129 00649 0.00 000000

See Reverse Side For Explanation Of Symbols And Abbreviations And Our Standard Terms And Conditions

Respectfully Submitted
Lancaster Laboratories, Inc.
Reviewed and Approved by:

Richard C. Entz, Group Leader
Misc. Organics/Ind. Hygiene

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Method Blank
Selected VOCs on charcoal tubes

*** BLANK INFORMATION ***
Matrix.....: Air/Charcoal Tube
Batch Number.....:
Injection number.....:
Analysis date.....:

Sample Information			
LLI Sample No.	Client Designation	Analysis Date	Analysis Time

Blank Contamination Information			
CAS Number	Compound	Blank Result	LOQ (mg)
	Acetone	ND	0.008
	Chlorobenzene	ND	0.014
	Chloroform	ND	0.022
	1,1-Dichloroethane	ND	0.013
	1,1-Dichloroethene	ND	0.012
	Ethyl benzene	ND	0.006
	Methyl ethyl ketone	ND	0.009
	Methyl isobutyl ketone	ND	0.009
	Methylene chloride	ND	0.022
	Tetrachloroethylene	ND	0.042
	Toluene	ND	0.008
	1,1,1-Trichloroethane	ND	0.022
	1,1,2-Trichloroethane	ND	0.022
	Trichloroethylene	ND	0.024
	Xylenes (sum of isomers)	ND	0.009

Comments:

LOQ = Limit of Quantitation
ND = None Detected
NR = Not Reported

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Where quality is a science.

Quality Control Summary
Surrogate Recovery
Selected VOCs on Charcoal Tubes

Batch Number:
SDG Number:

LLI Sample No.	Client Designation	Dilution Factor	Inject. No.	Surrogate Butanol #	Comment

Acceptance Criteria:	LOW	HIGH
Surrogate (n-Butanol)	85	125

Column to be used to flag recovery values
* Values outside QC limits

D Surrogates diluted out

Lancaster Laboratories
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Quality Control Summary
Matrix Spike\Matrix Spike Duplicate
Selected VOCs on Charcoal Tubes

Unspiked Sample Number :
Spiked Sample Number : Inj.:
Spiked Dup Sample Number: Inj.:
Batch Number: Inj.:
SDG Number : Date :

This MS/MSD applies to the following samples

Compound	Spike Added (mg)	Sample Concentration (mg)	MS Concentration (mg)	MS % REC	QC Limits REC	Comments
Acetone	0.033	ND	0.033	100	75 -125	
Chlorobenzene	0.100	ND	0.100	100	75 -125	
Chloroform	0.100	ND	0.100	100	75 -125	
1,1-Dichloroethane	0.100	ND	0.100	100	75 -125	
1,1-Dichloroethene	0.100	ND	0.100	100	75 -125	
Ethyl benzene	0.033	ND	0.033	100	75 -125	
Methyl ethyl ketone	0.033	ND	0.033	100	75 -125	
Methyl isobutyl ketone	0.033	ND	0.033	100	75 -125	
Methylene chloride	0.100	ND	0.100	100	75 -125	
Tetrachloroethylene	0.100	ND	0.100	100	75 -125	
Toluene	0.033	ND	0.033	100	75 -125	
1,1,1-Trichloroethane	0.100	ND	0.100	100	75 -125	
1,1,2-Trichloroethane	0.100	ND	0.100	100	75 -125	
Trichloroethylene	0.100	ND	0.100	100	75 -125	
Xylenes (sum of isomers)	0.033	ND	0.033	100	75 -125	

Compound	Spike Added (mg)	MSD Concentration (mg)		MSD % REC	QC Limits REC	% RPD	QC Limit RPD
Acetone	0.033	0.033		100	75 -125	0.0	15
Chlorobenzene	0.100	0.100		100	75 -125	0.0	15
Chloroform	0.100	0.100		100	75 -125	0.0	15
1,1-Dichloroethane	0.100	0.100		100	75 -125	0.0	15
1,1-Dichloroethene	0.100	0.100		100	75 -125	0.0	15
Ethyl benzene	0.033	0.033		100	75 -125	0.0	15
Methyl ethyl ketone	0.033	0.033		100	75 -125	0.0	15
Methyl isobutyl ketone	0.033	0.033		100	75 -125	0.0	15
Methylene chloride	0.100	0.100		100	75 -125	0.0	15
Tetrachloroethylene	0.100	0.100		100	75 -125	0.0	15
Toluene	0.033	0.033		100	75 -125	0.0	15
1,1,1-Trichloroethane	0.100	0.100		100	75 -125	0.0	15
1,1,2-Trichloroethane	0.100	0.100		100	75 -125	0.0	15
Trichloroethylene	0.100	0.100		100	75 -125	0.0	15
Xylenes (sum of isomers)	0.033	0.033		100	75 -125	0.0	15

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Initial Calibration
Selected VOCs on Charcoal Tubes

Calibration Date.....:

Instrument Identification..:

Laboratory Standard ID

This IC applies to samples:	Compound	DF 2000 STD 1	DF 400 STD 2	DF 100 STD 3	DF 40 STD 4	DF 20 STD 5	AVE RF	%RSD	%RSD QC Limit
	Acetone								25
	Chlorobenzene								25
	Chloroform								25
	1,1-Dichloroethane								25
	1,1-Dichloroethene								25
	Ethyl benzene								25
	Methyl ethyl ketone								25
	Methyl isobutyl ketone								25
	Methylene chloride								25
	Tetrachloroethylene								25
	Toluene								25
	1,1,1-Trichloroethane								25
	1,1,2-Trichloroethane								25
	Trichloroethylene								25
	p-Xylene								25
	m-Xylene								25
	o-Xylene								25

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Where quality is a science.

Quality Control Summary
Continuing Calibration
Selected VOCs on charcoal tubes

Calibration Date.....:

Instrument Identification..: Inj #..:

Continuing Calibration Date: Units: ug/mL

Compound	Reference Concentration	Continuing Calib Result	Acceptance Range 85%-	115%	Out of Range
Acetone	100.000		85.0	115.0	
Chlorobenzene	300.000		255.0	345.0	
Chloroform	300.000		255.0	345.0	
1,1-Dichloroethane	300.000		255.0	345.0	
1,1-Dichloroethene	300.000		255.0	345.0	
Ethyl benzene	100.000		85.0	115.0	
Methyl ethyl ketone	100.000		85.0	115.0	
Methyl isobutyl ketone	100.000		85.0	115.0	
Methylene chloride	300.000		255.0	345.0	
Tetrachloroethylene	300.000		255.0	345.0	
Toluene	100.000		85.0	115.0	
1,1,1-Trichloroethane	300.000		255.0	345.0	
1,1,2-Trichloroethane	300.000		255.0	345.0	
Trichloroethylene	300.000		255.0	345.0	
Xylenes	100.000		85.0	115.0	

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Where quality is a science.

Quality Control Summary
Method Blank
Phenol by HPLC

*** BLANK INFORMATION ***
Matrix.....:XAD-7 Tube
Batch Number.....:
Injection number.....:
Analysis date.....:

Sample Information				Blank Contamination Information			
LLI Sample No.	Client Designation	Analysis Date	Analysis Time	CAS Number	Compound	Blank Result	LOQ ppm
					Phenol	ND	0.2

Comments:

LOQ = Limit of Quantitation
ND = None Detected
NR = Not Reported

Lancaster Laboratories
Where quality is a science.

Quality Control Summary
Matrix Spike Duplicate

Unspiked Sample Number :
Spiked Sample Number : Inj.:
Spiked Dup Sample Number: Inj.:
Batch Number: Inj.:
SDG Number : Date :

This MS/MSD applies to the following samples	Compound	Spike Added (mg)	Sample Concentration (mg)	MS Concentration (mg)	MS % REC	QC Limits REC	Comments
	Phenol	0.240	ND	0.240	100	75 -125	

Compound	Spike Added (mg)	MSD Concentration (mg)		MSD % REC	QC Limits REC	% RPD	QC Limit RPD
Phenol	0.240	0.240		100	75 -125	0.0	15

APPENDIX D

ATEC ASSOCIATES, INC.

STANDARD OPERATING PROCEDURES FOR PARTICLE SIZE ANALYSIS

ATEC ASSOCIATES, INC.
STANDARD OPERATING PROCEDURE
PARTICLE SIZE ANALYSIS

1. **Parameter:** Soil Particle - size distribution

2. **Range of Measurement:** 0.0 - 100%

3. **Limit of Detection:** 0.0%

4. **Sample Matrix:** Soil

5. **Principle, Scope and Application:**

   Determine the particle size distribution in order to help classify the soil type and identify its properties.

6. **Interferences and Corrective Actions:**

   Equipment failure or technician error readjust, repair, or replace defective equipment and/or discuss error with the technician. Conduct duplicate test as necessary.

7. **Safety Precautions:**

   Wear safety glasses or gloves as needed for the contaminant present. Use alternate preparation methods if necessary.

8. **Sample Size, Collection, Preservation and Handling:**

   Sample size is dependent upon the largest diameter sediment grain in the soil to be analyzed. Each sample should be delivered/shipped in a sealed container (bag, jar, etc.) that is of sufficient size to contain the sample and will not permit the loss of any portion of the sample.

9. **Apparatus:**

- Balance sensitive to 0.01g,
- Mortor and rubber- tipped pestle,
- Stirring device "A" (per ASTM D-422; para. 3.2.1),
- Sedimentation cylinder (1000 ml),
- Soil Hydrometer 152-H (per ASTM D-422, para 3.3), and
- Sieves (per ASTM D-422; para. 3.6)

10. **Routine Preventative Maintenance:**

Balances are checked and serviced by a qualified service technician annually, and spot checked as needed. Sieves are checked with glass calibration spheres annually.

11. **Reagents & Calibration Standards:** None

12. **Calibration Procedures:** None

13. **Sample Preparation:**

Samples are air dried at room temperature then broken down to particle size using a mortor and rubber-tipped pestle.

14. **Analytical Measurement:** Not Applicable

15. **Flow Chart:** See Attached Exhibit

16. **Data Treatment:**

% Soil in Suspension	Diameter of Soil Particles
P = (Ra/W) X100	D = K x SQRT (L/T)
R = Hydrometer reading	K = Constant (Table 3; ASTM D-422)
A = Correction factor (Table 1, ASTM D-422)	
W = Oven dry soil weight	L = Effective depth (Table 2; ASTM D-422)
	T = Elapse time of reading
	SQRT = Square root

17. **Data Deliverables:**

a. Case narrative

b. Summary of all tests, and QC duplicate results (if required).

c. Copies of lab data sheets (if required)

d. Copies of any other pertinent sample documentation.

18. **Quality Control Requirement:**

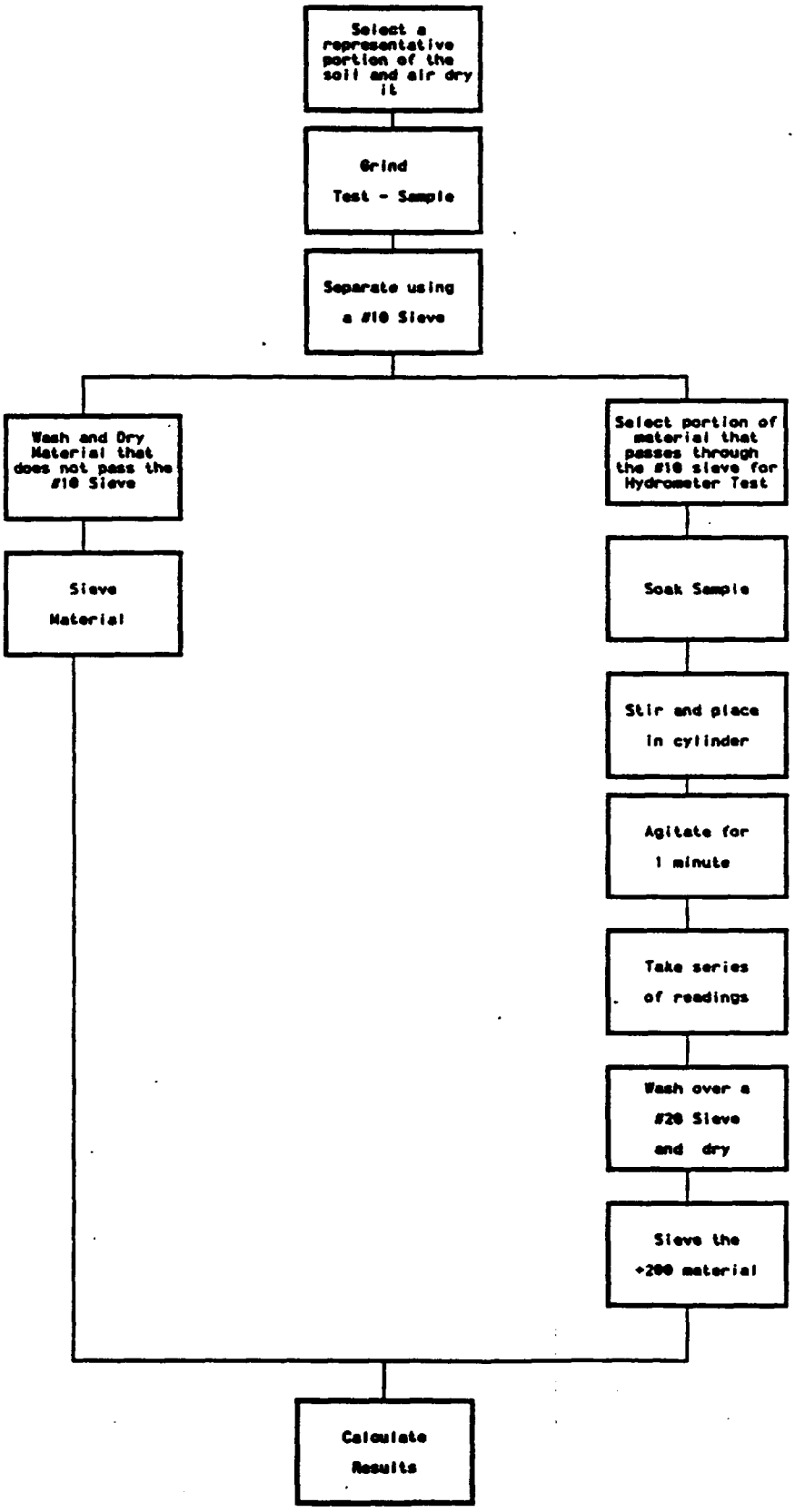
Duplicate test as requested.

19. **References:**

ASTM D-422-63

20. **Method Validation Data:** Not-Applicable

Particle Size Analysis Flow Chart



ATEC ASSOCIATES, INC.

SOILS LABORATORY

LABORATORY SAMPLE CUSTODY

Upon receipt, the chain-of-custody (if possible) is signed by the lab supervisor, or technician, that receives the samples. The samples are then logged, and assigned a consecutive sample log number. A work order is prepared for the sample, or group of samples, and the test(s) that are to be conducted on each sample is indicated. If a sample requires both physical and chemical tests, a separate chemistry lab work order is prepared and a representative portion of the sample is obtained and taken to the chemistry lab. All work orders, sample tags, lab sheets, etc. must have the appropriate sample log numbers on them. The unused portions of the samples are maintained in the laboratory for 30 days after the test is completed unless other arrangements are made by the client.

ATEC Associates, Inc.

LAB WORK ORDER
ENGR. & DRILLING DIV.

JOB NO. _____ DATE ASSIGNED _____

JOB NAME _____ DATE REQUIRED _____

CLIENT _____

COMPUTER → BILLING CODE																					
														PERM.		PROCTOR				OTHER	
BORING	SAMPLE	DEPTH	EXTRUDE	MC	DENSITY	WASHED SIEVE	HYDR.	-200	PI	CONSOL.	UNCONF.	UU*	CU*	FALLING HEAD	TRIAX.	STD	MOD	CBR	LOI		

(* GIVE NO. OF PTS.)

POSSIBLE HAZARDOUS MATERIALS ☐ YES ☐ NO

SPECIAL INSTRUCTIONS: _____

_____

_____

_____

_____ ASSIGNED BY: _____

ATEC Associates, Inc.

LAB WORK ORDER
MATERIALS DIVISION

JOB NO. _____ DATE ASSIGNED _____

JOB NAME _____ DATE REQUIRED _____

LOCATION _____

CLIENT _____

POSSIBLE HAZARDOUS MATERIALS ☐ YES ☐ NO

SAMPLE (CIRCLE)

SOIL AGGREGATE ASHALT (CORE or BULK) CONCRETE CORES

BRICK FIREPROOFING ROOFING CEMENT

OTHER: _____

NO. OF SAMPLES: _____

	COMPUTER→ BILLING CODE												
SAMPLE LOG NO.	SAMPLE SOURCE AND DESCRIPTION	PROCTOR		SIEVE		EXTRACTION & GRADATION	DENSITY	MARSHALL STABILITY & FLOW	COMPRESSIVE STRENGTH	OTHER			
		STD	MOD	-200 ONLY	WASHED								

SAMPLE LOCATION: ☐ LIBRARY ☐ PROCTOR ROOM ☐ MAIN LAB ☐ CONCRETE LAB

SPECIAL INSTRUCTIONS: _____

ASSIGNED BY: _____

PROJ. NO.:

CLIENT

SAMPLERS: *(Signature)*

SAMPLING METHOD

SAMPLE I.D. NO.	DATE	TIME	COMPOSITE	GRAB	WATER	SOIL		FILTERED	ACIDIFIED	ICED		NUMBER OF CONTAINERS	LAB I.D. NUMBER	VOLATILE ORGANICS	BTX & E	TOTAL HYDROCARBONS	PCB'S	E.P. TOXIC METALS	TOTAL METALS (8)	IGNITABILITY			SAMPLE LOCATION / REMARKS

LABORATORY ANALYSIS

ATEC Environmental Services
Division of ATEC Associates, Inc.
5150 East 65th Street
Indianapolis, Indiana 46220-4871
(317) 849-4990, FAX # (317) 849-4278

Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date / Time	Received by: *(Signature)*

Relinquished by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Project Manager / Phone #:

SECTION NO.: 7
REVISION NO.: 0
DATE: 8/28/96
PAGE 4 OF 6

IN HOUSE CHAIN OF CUSTODY

ATEC LAB NO._____

DATE RECEIVED_____ CLIENT JOB NO._____

MATRIX_____ TOTAL # OF SAMPLES_____
EXTRACTION REQUIRED (Y OR N)___ ORGANIC Y/N____ INORGANIC Y/N_____

SAMPLES PLACED IN COOLER: DATE_____ TIME_____ by_____

RELINQUISHED BY:	RECEIVED BY:(AREA)	DATE/TIME

SAMPLES/EXTRACTS PLACED IN POST ANALYSIS STORAGE BY_____

DATE_____ TIME_____

SAMPLES/EXTRACTS DISPOSED OF BY_____

DATE_____ TIME_____

METHOD OF DISPOSAL_____

APPENDIX E

FIELD EQUIPMENT
STANDARD OPERATING PROCEDURES

APPENDIX E

TABLE OF CONTENTS

E.1 Field Measurements of pH

E.2 Field Measurements of Specific Conductance and Temperature

APPENDIX E.1

FIELD MEASUREMENTS OF pH

Field Measurements of pH

Method: Electrometric measurement of pH

Sensitivity: 0.1 pH Unit

Optimum Range: 1 - 12 pH Units

Sample Measurement: On-site upon sample collection

Reagents and Apparatus:

1. pH meter (Orien Model 211 min-pH meter or equivalent)
2. Combination electrodes
3. Beakers or plastic cups
4. pH buffer solutions, pH 4, 7, and 10
5. Deionized water in squirt bottle
6. All glassware shall be soap and water washed, followed by two hot water rinses and two deionized water rinses.

Procedures

A. **Calibration:**

1. Place electrode in pH 7 buffer solution.
2. After allowing several minutes for meter to stabilize, turn calibraiton dial until a reading of 7.00 is obtained.
3. Rinse electrode with deionized water and place in pH 4 or pH 10 buffer solution.

   NOTE: When calibrating the meter for samples with pH < 8.00, use buffers 7 and 4; and, for samples with pH > 8.00, use buffers pH 7 and 10.

4. Wait several minutes and then turn slope adjustment dial until a reading of 4.00 or 10.00 is obtained.
5. Rinse electrode with deionized water and place in buffer pH 7. If meter

reading is not 7.00, repeat steps 2 - 5.

B. Sample Measurements:

1. calibrate the meter according the calibraiton procedure.

2. Pour sample into a clean beaker or plastic cup.

3. Place the electrode in the sample solution. Make sure the white KCl junction on side of electrode is in solution. The level of electrode solution shall be one inch above sample to be measured.

4. Record the reading along with the temperature of the solution in the field/laboratory logbook.

5. Rinse electrode with deionized water between samples. Recheck calibration with pH 7 buffer solution after every 5 samples.

6. Repeat step 2-5 for each sample.

Quality Control:

1. Recheck calibration with pH 7 buffer solution after every 5 samples. The reading shall not exceed 7.00 +0.01 pH unit.

2. If, during meter calibration or calibration check, the meter fail to read 4.00 or 10.00, something may be wrong with the electrode. The cause shall be investigated and corrected. If problem lies in the electrode, the electrode shall be replaced.

3. pH is temperature dependent analysis. Therefore the temperature of buffer and smaples shall be recorded. The difference of temperature between the buffer solutions and samples shall be within about 2^0C. For refrigerated or cooled samples, refrigerated or cooled buffer solutions shall be used to calibrate the meter.

4. When not in use, the electrodes shall be stored in pH 4 buffer.

5. Weak organic acids, inorganic salts, and oil and grease interferes the pH measurements. If oil and grease are visible, note it on the field logbook and data sheet. Clean the electrode with soap and water,, followed with 10% HCl. Then recalibrate the meter.

6. Before going into the field:

   The following preparations shall be performed:

   a. Check batteries;

b. Calibration at pH 7 and 4 to check the electrode.
c. Obtain fresh buffer solutions.

7. Following field measurements:

   a. Report any problems;
   b. compare with previous data;
   c. Clean all dirt off meter and inside case;
   d. Make sure electrode is stored in pH 4 buffer.

References:

1. EPA Method No.150.1, "Methods for Chemical Analysis of Water and Wastes" 1979, Revised 1983.

APPENDIX E.2

FIELD MEASUREMENTS OF SPECIFIC CONDUCTANCE AND TEMPERATURE

Field Measurements of Specific Conductance and Temperature

Method: Specific Conductance, umhos at 25°C

Detection Limits: 1 umho/cm at 25°C

Optimum Ranges: 0.1 - 100,000 umhos/cm

Sample Measurement: Measure on-site upon sample collection

Reagents and Apparatus:

1. Conductivity meter (YSI or equivalent) and electrodes
2. Deionized water in squirt bottle.
3. Stock potassium chloride soultion, 1.00 N : Dissolve 74.555 g of KCl in Milli-Q water and dilute to 1,000 ml in a 1-liter volumetric flask.
4. Standard potassium chloride solution, 0.0100 N : Pipet 10 ml of stock solution into 1,000 ml volumetric flask and dilute with milli-Q water to the mark.

Procedures:

1. With mode switch at off position, check meter zero. If it can not be zero, use meter screw and adjust to zero.
2. Plug probe into jack on side of meter.
3. Turn mode switch to red line, and turn red line knob until need aligns with red line on dial. Change batteries if it can not be aligned.
4. Totally immerse probe in sample. Note: Do NOT allow the probe to touch the sample container.
5. Turn mode switch to appropriate conductivity scale, X100, X10, or X1. Use a scale that will give a mid-range output on the meter.
6. Wait for the needle to stabilize (about 15 seconds) and record conductivity multiplying by scale setting in field logbook.
7. While gently agitating the probe, take sample temperature (°C) and record in field logbook.

8. Rinse probe with deionized water.

9. Calculate specific conductivity using the following equation:

$$G_{25} = \frac{G_T}{\{ 1 + 0.02\ (T-25) \}}$$

Where:

G_{25} = conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

G_T = Conductivity of sample at temperature T°C, umhos/cm

10. Record specific conductivity and temperature in the field logbook, and on appropriate data sheet. Report results for the standard solution with each data set.

11. Record on field logbook which meter and probe were used.

Quality Control:

1. After use, the meter must be wiped clean as possible.

2. After returning to the laboratory, compare results with previous data, and report any problems encountered to the lab personnel.

3. Recheck calibration after every 5 samples.

References:

1. EPA Method No.120.1, "Methods for Chemical Analysis of Water and Wastes." 1979, revised 1983.

2. Standard Methods, 15th Edition.