

**APPENDIX G**

**ANALYTICAL PROCEDURES FOR  
INCHCAPE TESTING SERVICES ENVIRONMENTAL LABORATORIES  
SAN JOSE FACILITY**

**6.19 Method 8260A - Volatile Organic Compounds By Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique (Sept. 1994)**

**Approval Signatures:**

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**1.0 SCOPE AND APPLICATION**

1.1 Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. The following compounds may be determined by this method:

<b>ANALYTE</b>	<b>CAS #</b>
Acetone	67-64-1
Benzene	71-43-2
Bromobenzene	108-86-1
Bromochloromethane	74-97-5
Bromodichloromethane	75-27-4
Bromoform	75-25-2
Bromomethane	74-83-9
2-Butanone	78-93-3
n-Butylbenzene	104-51-8
sec-Butylbenzene	135-98-8
tert-Butylbenzene	98-06-6
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chlorodibromomethane	124-48-1
Chloroethane	75-00-3
Chloroform	67-66-3
1-Chlorohexane	544-10-5
Chloromethane	74-87-3
2-Chlorotoluene	95-49-8
4-Chlorotoluene	106-43-4
1,2-Dibromo-3-chloropropane	96-12-8
1,2-Dibromoethane	106-93-4
Dibromomethane	74-95-3
1,2-Dichlorobenzene	95-50-1

1,3-Dichlorobenzene	541-73-1
1,4-Dichlorobenzene	106-46-7
Dichlorodifluoromethane	75-71-8
1,1-Dichloroethane	75-34-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethene	75-35-4
cis-1,2-Dichloroethene	156-59-2
trans-1,2-Dichloroethene	156-60-5
1,2-Dichloropropane	78-87-5
1,3-Dichloropropane	142-28-9
2,2-Dichloropropane	594-20-7
1,1-Dichloropropene	563-58-6
cis-1,3-Dichloropropene	10061-01-5
trans-1,3-Dichloropropene	10061-02-6
Ethylbenzene	100-41-4
Hexachlorobutadiene	87-68-3
2-Hexanone	591-78-6
Isopropylbenzene	98-82-8
p-Isopropyltoluene	99-87-6
Methylene chloride	75-09-2
4-Methyl-2-pentanone	108-10-1
Naphthalene	91-20-3
n-Propylbenzene	103-65-1
Styrene	100-42-5
1,1,1,2-Tetrachloroethane	630-20-6
1,1,2,2-Tetrachloroethane	79-34-5
Tetrachloroethene	127-18-4
Toluene	108-88-3
1,2,3-Trichlorobenzene	87-61-6
1,2,4-Trichlorobenzene	120-82-1
1,1,1-Trichloroethane	71-55-6
1,1,2-Trichloroethane	79-00-5
Trichloroethene	79-01-6
Trichlorofluoromethane	75-69-4
Trichlorotrifluoroethane	76-13-1
1,2,3-Trichloropropane	96-18-4
1,2,4-Trimethylbenzene	95-63-6
1,3,5-Trimethylbenzene	108-67-8
Vinyl acetate	108-05-4
Vinyl chloride	75-01-4
Total Xylenes	1330-20-7
o-Xylene	95-47-6
m-Xylene	108-38-3
p-Xylene	106-42-3

## **INSTRUMENTATION: GC/MS**

1.2 Method 8260 can be used to quantitate most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique. However, for the more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency. Such compounds include low-molecular-weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides. See Tables 1 and 2 for lists of analytes and retention times that have been evaluated on a purge-and-trap GC/MS system. Also, the method detection limits for 25 mL sample volumes are presented.

1.3 The estimated quantitation limit (EQL) of Method 8260 for an individual compound is approximately 5 µg/Kg (wet weight) for soil/sediment samples, 0.5 mg/Kg (wet weight) for wastes, and 5 µg/L for ground water (see Table 3). EQLs will be proportionately higher for sample extracts and samples that require dilution or reduced sample size to avoid saturation of the detector.

1.4 Method 8260 is based upon a purge-and-trap, gas chromatographic/mass spectrometric (GC/MS) procedure. This method is restricted to use by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

## **2.0 SUMMARY OF METHOD**

2.1 The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method. Purged sample components are trapped in a tube containing suitable sorbent materials. When purging is complete, the sorbent tube is heated and backflushed with helium to desorb trapped sample components. The analytes are desorbed directly to a large bore capillary or cryofocused on a capillary precolumn before being flash evaporated to a narrow bore capillary for analysis. The column is temperature programmed to separate the analytes which are then detected with a mass spectrometer (MS) interfaced to the gas chromatograph. Wide bore capillary columns require a jet separator, whereas narrow bore capillary columns can be directly interfaced to the ion source.

2.2 If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in a solvent (e.g. methanol) to dissolve the volatile organic constituents. A portion of the solution is combined with organic-free reagent water in the purge chamber. It is then analyzed by purge-and-trap GC/MS following the normal water method.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a jet separator or a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard with a five-point calibration curve.

2.4 The method includes specific calibration and quality control steps that replace the general requirements in SW-846 Method 8000.

### 3.0 INTERFERENCES

3.1 Major contaminant sources are volatile materials in the laboratory and impurities in the inert purging gas and in the sorbent trap. The use of non-polytetrafluoroethylene (PTFE) thread sealants, plastic tubing, or flow controllers with rubber components should be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should change the purge gas source and regenerate the molecular sieve purge gas filter. Subtracting blank values from sample results is not permitted. If reporting values not corrected for blanks result in what the laboratory feels is a false positive for a sample, this should be fully explained in text accompanying the uncorrected data.

3.2 Interfering contamination may occur when a sample containing low concentrations of volatile organic compounds is analyzed immediately after a sample containing high concentrations of volatile organic compounds. The preventive technique is rinsing of the purging apparatus and sample syringes with two portions of organic-free reagent water between samples. After analysis of a sample containing high concentrations of volatile organic compounds, one or more calibration blanks should be analyzed to check for cross contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds or high concentrations of compounds being determined, it may be necessary to wash the purging device with a soap solution, rinse it with organic-free reagent water, and then dry the purging device in an oven at 105°C. In extreme situations, the whole purge and trap device may require dismantling and cleaning. Screening of the samples prior to purge and trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples. Screening may be accomplished with an automated headspace technique.

3.2.1 The low purging efficiency of many analytes from a 25 mL sample often results in significant concentrations remaining in the sample purge vessel after analysis. After removal of the analyzed sample aliquot and three rinses of the purge vessel with analyte-free water. To confirm the absence of carryover it may be necessary to run an instrument blank(s).

3.3 Special precautions must be taken to analyze for methylene chloride. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride. Otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all gas chromatography carrier gas lines and purge gas plumbing should be constructed from stainless steel or copper tubing. Laboratory clothing worn by the analyst should be clean since clothing previously exposed to methylene chloride fumes during liquid/liquid extraction procedures can contribute to sample contamination.

3.4 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip blank prepared from organic-free reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Storage blanks prepared from organic-free water are stored in the sample storage areas and analyzed on a weekly basis.

3.5 Use of sensitive mass spectrometers to achieve lower detection levels will increase the potential to detect laboratory contaminants as interferences.

3.6 If hexadecane is added to samples or petroleum samples are analyzed, some chromatographic peaks will elute after the target analytes. The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are volatilized.

#### **4.0 APPARATUS AND MATERIALS**

4.1 Purge-and-trap device - The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. The Dynatech PTA-30 S/W and Dynatech Archon 5100 combined with the Tekmar LSC 2000 are purge and trap units capable of purging water and soil samples.

4.1.1 Both the Dynatech PTA-30 S/W and Archon purge water samples introduced in headspace-free 40 mL VOA vials. A standard 5 mL sample volume is transferred to a conventional purge chamber on the Tekmar LSC 2000. Both autosampler units are capable of performing dilutions ranging from 2X to 10X. Larger sample volumes can be purged from the purge chamber associated with each unit. A standard sample purge chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Larger chambers are available for purging 25 mL samples. Alternate sample purge devices may be utilized, provided equivalent performance is demonstrated. As described above, the Dynatech autosamplers purge water samples directly from the 40 mL VOA vials in which the

samples are collected. The samples are purged by the insertion of a pair of concentric needles through the VOA vial septum. The Dynatech PTA-30 S/W and Archon 5100 units use helium to pressurize the VOA bottle and push an aliquot of sample along a transfer line into the Tekmar LSC 2000 purging chamber, where it is purged in normal fashion.

4.1.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. Two types of trap are used at the laboratory. Starting from the inlet, the first analytical trap used is composed of 16 cm Tenax polymer and 7.7 cm activated charcoal (Supelco part no. 2-1061). The second trap used is the Supelco VOCARB 3000 composed of Carboxen B/ Caboxen 1000 and 1001 (Part #2-1066). Before initial use, the trap should be conditioned at 270°C for 60 minutes followed by an extended desorb and bake cycle.

#### 4.2 Gas chromatograph/mass spectrometer system.

4.2.1 Gas chromatograph - Hewlett Packard 5890 Series I and Series II chromatographs, equipped with packed injection ports adapted with special low volume inserts (O.I. Analytical). The low volume inserts include a nut with septum that allows for manual injection into the injection port.

4.2.2 Column - DB-624, 75 meter, 0.53 mm i.d., 0.5  $\mu$ m thickness coating.

4.2.3 Mass spectrometer - Hewlett Packard 5971A and 5972 MSD (Mass Selective Detectors) capable of scanning from 35-280 m/z every second, using 70 volts (nominal) electron energy in the electron impact mode and producing a mass spectrum that meets all the criteria in Table 3 when 50 ng of 4-bromofluorobenzene (BFB) are injected through the gas chromatograph inlet.

4.2.4 GC/MS interface - Because megabore columns are used for analysis (0.53 mm i.d.) a glass jet separator (manufactured and supplied as standard equipment by Hewlett Packard) is used to reduce the total He<sub>2</sub> carrier gas flow being introduced into the MSD. The column eluant is transferred from the jet separator to the MSD via a heated, deactivated transfer line.

4.3 Data system - A DOS based PC is used for instrument control and data acquisition. A network version of Chemserver (Hewlett Packard) is used to set up run sequences, control the purge and trap device and control the GC and MSD. Target Thru-Put and Envisions is used to acquire, process and report mass spectral data. This computer system allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. This system allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). This software also allows integrating the abundances in any EICP

between specified time or scan number limits. The most recent version of the EPA/NIST Mass Spectral Library which contains 75,000 entries is loaded on the system.

4.4 Microsyringes - 10, 25, 100, 250, 500 and 1000  $\mu$ L.

4.5 Syring valve - Two-way with Luer ends.

4.6 Syringes - 5, 10 or 25 mL, gas-tight with shut off valve.

4.7 Balance - top-loading, 0.1 g.

4.8 Glass vials - 40 mL with Teflon-lined screw caps.

4.9 Volumetric flasks, Class A - 10 and 100 mL, with ground glass stoppers.

4.10 Spatula - Stainless steel.

## 5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.1.1 Solvent lot checks - Solvents such as methanol must be checked for purity on a lot basis. To check a solvent, it must be prepared in a similar manner to that in which it will be used and analyzed as a sample. The check sample must be free of all target compounds, and TIC compounds should be at a minimum. Store all solvent lot check chromatograms in the notebook located by the instruments.

5.2 Organic-free reagent water - This water is obtained from the Inorganics Department Millipore Milli-Q System and prepared by boiling for 15 minutes and then purging the water with a fine stream of helium. The water must be continuously sparged with helium through its entire use. Water should be prepared fresh daily.

5.3 Stock solutions - Stock solutions are prepared from certified solutions purchased from reputable vendors, such as Supelco and Restek, accompanied with certificates of analysis and documentation materials. The documents are stored in a binder in the QA/QC Department. When stock solutions are to be used to prepare dilute working solutions the following guidelines are to be followed.

5.3.1 Allow the ampule or sealed vial of stock solution to warm to room temperature before opening. Perform all calculations and assemble the necessary volumetric flasks, (lot checked) purge and trap methanol and microliter syringes before opening the stock solution.

5.3.2 Rinse the volumetric flask with methanol and discard the methanol three times. Fill the volumetric flasks with enough methanol such that when the appropriate amount of stock solution(s) is added there will be less than 1 mL volume to bring the flask to volume.

5.3.3 Pick microliter syringes that have a total volume close to the volume you wish to measure. Example: If a 20 uL is required, use a 25 uL syringe, not a 50 uL syringe. Rinse all microliter syringes 3 times with clean methanol and discard the methanol. Confirm that the syringes are clean, have no burrs on the needles, and do not trap bubbles when inverted.

5.3.4 Open the stock solution vial or ampule. Note: Go through the following steps completely, one vial or ampule at a time. Draw a small amount of the stock standard into the syringe and then discard. Fill the syringe beyond the volume required, then pull in a small bubble of air. Invert the syringe, tap lightly to force all bubbles to the needle end of the syringe. Slowly adjust the syringe to the appropriate volume while holding a Kimwipe to the needle orifice, this allows the bubble and a small amount of liquid to be purged from the syringe. Wipe the needle of excess solution and place the needle into the volumetric flask below the level of methanol and expel the stock solution into the flask. Do not "pump" the syringe.

5.3.5 Bring the volumetric flask to volume with methanol and stopper. Invert the flask and rotate several times so that the bubble of air circles the bottom of the upended flask. Right the flask. Repeat the last two steps twice more. Transfer the freshly prepared stock solution into a Teflon-capped container, which has little headspace. Discard the original vial or ampule and its remaining contents. Under no circumstances are stock solutions to be kept and used at a later time after they have been opened.

5.4 All prepared dilute solutions in methanol must be discarded after three months. Gasses and other reactive components such as 2-chloroethylvinyl ether will degrade more quickly if allowed to warm often. Monitor these components and replace more frequently if required.

5.5 Surrogate standards - The following may be used as surrogates: toluene-d8, 4-bromofluorobenzene, 1,2-dichloroethane-d4 and dibromofluoromethane. These are purchased as a mix from Restek. A stock surrogate solution in methanol should be prepared as described in Section 5.3, and a surrogate standard spiking solution should be prepared from the stock at a concentration of 25 µg/mL in methanol. Each sample undergoing GC/MS analysis must be spiked with 10 µL of the surrogate spiking solution prior to analysis.

**5.6 Internal standards** - The internal standards used are pentafluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene-d4 and chlorobenzene-d5. These are purchased as a mix from Restek. Prepare internal standard stock and secondary dilution standards in methanol using the procedures described in Sections 5.3 and 5.4. It is recommended that the secondary dilution standard should be prepared at a concentration of 25 µg/mL for soil analyses and 250 µg/mL for waters of each internal standard compound. Addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be the equivalent of 50 µg/L. The PTA -30 S/W and Archon automatically dispense internal standard solution into the samples by a syringe assembly which uses a sample loop of known volume. Each PTA-30 S/W and Archon must be individually calibrated to determine the exact volume of the sample loop. Calibration of the loop is done by analyzing aliquots of known volume (0.1, 0.5, 1.0, 1.5 and 2.0 µL) of internal standard solution in 5 mL blank samples. The abundance of 1,4-difluorobenzene is determined and is plotted versus the volume of the internal standard added to the blanks. Having the autosampler dispense internal standard solution in 5 mL blank samples, five water blanks are analyzed. The volume of sample loop is then determined by the average abundance of the blanks with unknown volume of internal standard added from the plot of abundance versus the known volume of internal standard.

**5.7 4-Bromofluorobenzene (BFB) standard** - A stock solution of BFB is purchased from Restek. A standard solution containing 25 ng/µL of BFB in methanol should be prepared.

**5.8 Calibration standards** - Calibration standards at a minimum of five concentrations should be prepared from the secondary dilution of stock standards (see Sections 5.3 and 5.4). Prepare these solutions in organic-free reagent water. Routinely, the concentrations prepared are 10, 20, 50, 100 and 200 µg/L for water or µg/Kg for soil. These levels may have to be adjusted to meet requirements for lower reporting limits. The low level standard is at a concentration equivalent to the reporting limit. The remaining concentrations should correspond to the expected range of concentrations found in real samples, but should not exceed the working range of the GC/MS system. Each standard should contain each target analyte. Calibration standards in reagent water must be disposed of within 24 hours of preparation.

**5.9 Matrix spiking standards** - Matrix spiking standards should be prepared from volatile organic compounds which will be representative of the compounds being investigated. The compounds are 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The standard is purchased as a mix from Restek and is prepared in methanol, with each compound present at a concentration of 25 µg/mL.

**5.10** Great care must be taken to maintain the integrity of all standard solutions. It is recommended that all standards in methanol be stored at -10°C to -20°C in screw-cap amber bottles with Teflon liners.

5.11 Methanol, Purge-and-trap quality. All lots of methanol must be tested as described in section 5.1.1.

## 6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 Samples should be analyzed as soon as possible after receipt. Samples should be stored in an area free from contamination and at a temperature of  $4\pm 2^{\circ}\text{C}$ . Maximum holding times for water and soil samples are 14 days from the time of sampling. Water samples should be preserved with hydrochloric acid to a pH of less than 2. Sample preservation should be checked at the time of analysis and documented in the Instrument Injection Logbook.

## 7.0 PROCEDURE

7.1 The only time direct injection is to be used is in the injection of the tuning compound, BFB, at the start of the calibration period.

7.2 Initial calibration for purge-and-trap procedure.

7.2.1 Recommended GC/MS operating conditions.

Electron energy	70 volts (nominal)
Mass range	35-280 amu
Scan time	1 scan/ sec
Initial column temperature	35°C
Initial column holding time	2 minutes
Column temperature program	6°C/minute to 100°C, hold for 2 min.
Final column temperature	10°C/min to 180°C.
Final column holding time	1.5 minutes
Injector temperature	200-225°C
Source temperature	The source temperature is kept at 140°C convectionally from the transfer line.
Transfer line temperature	250-300°C
Carrier gas	Helium at total column flow of 30 mL/min.

7.2.2 Each GC/MS system must be hardware tuned to meet the criteria in Table 4 for a 50 ng injection or purging of 4-bromofluorobenzene (1 uL injection of the BFB standard). Analyses must not begin until these criteria are met.

7.2.3 Assemble a purge-and-trap device that meets the specification in Section 4.11. Condition the trap at 180°C for 30 to 60 minutes in the purge mode with an inert gas flow of at least 20 mL/min. Prior to use, condition the trap daily by performing one or more blank purge and desorb/bake cycles.

7.2.4 Connect the purge-and-trap device to a gas chromatograph.

7.2.5 Prepare the final solutions containing the required concentrations of calibration standards, including surrogate standards, directly in the purging device (use freshly prepared stock solutions when preparing the calibration standards for the initial calibration). Add 5.0 mL of organic-free reagent water to the sample syringe. Next, using a 10  $\mu$ L or 25  $\mu$ L microsyringe equipped with a long needle (Section 4.4), take a volume of the secondary dilution solution containing appropriate concentrations of the calibration standards (Section 5.8). Add the aliquot of calibration solution directly to the organic-free reagent water in the syringe by inserting the needle through the Luer-lock tip. When discharging the contents of the microsyringe, be sure that the end of the syringe needle is well beneath the surface of the organic-free reagent water. Similarly, add 10  $\mu$ L of the internal standard solution (Section 5.6). Close the 2 way syringe valve at the sample inlet.

7.2.5.1 Another method of preparing the standards for purging is to perform tertiary dilutions of stock calibration standards in volumetric flasks. Once the dilution has been performed, a 5 mL aliquot of the working standard can be decanted into a sample syringe and loaded into the purge vessel after the addition of internal standards.

7.2.6 Carry out the purge-and-trap analysis procedure as described in Section 7.4.1.

7.2.7 Tabulate the area response of the characteristic ions (see Table 5) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured (Section 7.5.2). The RF is calculated as follows:

$$RF = (A_{(x)}C_{(is)}) / (A_{(is)}C_{(x)})$$

where:

$A_{(x)}$  = Area of the characteristic ion for the compound being measured.

$A_{(is)}$  = Area of the characteristic ion for the specific internal standard.

$C_{(is)}$  = Concentration of the specific internal standard.

$C_{(x)}$  = Concentration of the compound being measured.

7.2.8 The average RF must be calculated and recorded for each compound using the five RF values calculated for each compound from the initial calibration curve. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. These compounds are used to

check compound instability and to check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

7.2.8.1 Chloromethane - This compound is the most likely compound to be lost if the purge flow is too fast.

7.2.8.2 Bromoform - This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve bromoform response.

7.2.8.3 1,1,2,2-Tetrachloroethane and 1,1-dichloroethane - These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

7.2.9 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for all compounds. The percent RSD is calculated as follows:

$$\%RSD = (SD / \overline{RF}_x) \times 100$$

where:

$\%RSD$  = relative standard deviation.

$\overline{RF}_x$  = mean of 5 initial RFs for a compound.

SD = standard deviation of the 5 initial RFs for a compound.

The %RSD should be less than 15 percent for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30%. The CCCs are:

1,1-Dichloroethene  
Chloroform  
1,2-Dichloropropane  
Toluene  
Ethyl benzene  
Vinyl chloride

7.2.9.1 If a %RSD greater than 30% is measured for any CCC, then corrective action to eliminate a system leak and/or column reactive sites is required before reattempting calibration.

7.2.10 Linearity - If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio ( $A/A_{is}$ ) versus concentration using first or higher order

regression fit of the five calibration points. The analyst should select the regression order which introduces the least calibration error into the quantitation. Please note that this may be limited to first order by contract or project requirements. The use of calibration curves is a recommended alternative to average response factor calibration, and a useful diagnostic of standard preparation accuracy and absorption activity in the chromatographic system.

7.2.11 These curves are verified each shift by purging a performance standard. Recalibration is required only if calibration and ongoing performance criteria cannot be met.

### 7.3 GC/MS Calibration Verification

7.3.1 Prior to the analysis of samples, inject or purge 50 ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 4 before sample analysis begins. These criteria must be demonstrated each 12 hour shift.

7.3.2 The initial calibration curve (Section 7.2) for each compound of interest must be checked and verified once every 12 hours during analysis with the introduction technique used for samples. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS by checking the SPCC (Section 7.3.3) and CCC (Section 7.3.4).

7.3.3 System Performance Check Compounds (SPCCs) - A system performance check must be made each 12 hours. If the SPCC criteria are met, a comparison of relative response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

7.3.3.1 The minimum relative response factors (RRF) for volatile SPCCs are as follows:

<b>Compound</b>	<b>Min. RRF</b>
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	>0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

7.3.4 Calibration Check Compounds (CCCs) - After the system performance check is met, CCCs listed in Section 7.2.9 are used to check the validity of the initial calibration. Calculate the percent drift using the following equation:

$$\% \text{Drift} = (C_{(n)} - C_{(c)}) / C_{(n)} \times 100$$

where:

$C_{(n)}$  = Calibration Check Compound standard concentration.

$C_{(c)}$  = Measured concentration using selected quantitation method.

If the percent drift for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met for any one CCC, corrective action must be taken. All other compounds of interest should have a % Drift of less than 50%. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five point calibration must be generated. This criterion must be met before quantitative sample analysis begins. If the CCCs are not required analytes by the permit, then all required analytes must meet the 20% Drift criterion.

7.3.5 The internal standard responses and retention times in the check calibration standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check (12 hrs), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a factor of two (-50 to +100%) from the last daily calibration check standard, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is necessary.

## 7.4 GC/MS Analysis

### 7.4.1 Water samples

7.4.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Screening is performed at the laboratory by automated headspace analysis using a gas chromatograph (GC) equipped with an FID.

7.4.1.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

7.4.1.3 Set up the GC/MS system as outlined in Section 7.2.1.

7.4.1.4 Confirm that the BFB tuning criteria and GC/MS calibration verification criteria were met (Section 7.3) before analyzing samples.

7.4.1.5 Adjust the purge gas (helium) flow rate to 40 mL/min on the purge-and-trap device. Optimize the flow rate to provide the best response for chloromethane and bromoform, if these compounds are target compounds. Excessive flow rate reduces chloromethane response, whereas insufficient flow reduces bromoform response (see Section 7.2.8).

7.4.1.6 Under normal circumstances, water samples are sampled directly from the 40 mL VOA bottles in which they were collected. When this occurs, the autosampler automatically spikes internal standards and surrogates into the sample. Some circumstances will require the manual loading of a water sample directly into the purging chamber. To do this, remove the plunger from a 5 mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20 mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

7.4.1.7 The following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.4.1.7.1 Dilutions may be made in volumetric flasks (10 to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

7.4.1.7.2 Calculate the approximate volume of organic-free reagent water to be added to the volumetric flask selected and add slightly less than this quantity of organic-free reagent water to the flask.

7.4.1.7.3 Inject the proper aliquot of sample from the syringe prepared in Section 7.4.1.6 into the flask. Aliquot of less than 1 mL are not recommended. Dilute the sample to the mark with organic-free reagent water. Cap the flask, invert, and shake three times. Repeat above procedure for additional dilutions.

7.4.1.7.4 Fill a 5 mL syringe with the diluted sample as in Section 7.4.1.6.

7.4.1.8 Add 10.0  $\mu\text{L}$  of surrogate spiking solution (Section 5.5) and 10  $\mu\text{L}$  of internal standard spiking solution (Section 5.6) through the valve bore of the syringe; then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution. The addition of 10  $\mu\text{L}$  of the surrogate spiking solution to 5 mL of sample is equivalent to a concentration of 50  $\mu\text{g/L}$  of each surrogate standard.

7.4.1.9 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

7.4.1.10 Close both valves and purge the sample for  $10.0 \pm 0.1$  minutes at ambient temperature.

7.4.1.11 At the conclusion of the purge time, attach the trap to the chromatograph, adjust the device to the desorb mode, and begin the gas chromatographic temperature program and GC/MS data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to  $180^\circ\text{C}$  while backflushing the trap with inert gas between 20 and 60 mL/min for 4 minutes. If this rapid heating requirement cannot be met, the gas chromatographic column must be used as a secondary trap by cooling it to  $30^\circ\text{C}$  (or subambient, if problems persist).

7.4.1.12 While the trap is being desorbed into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of organic-free reagent water (or methanol followed by organic-free reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

7.4.1.13 After desorbing the sample for 4 minutes, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 seconds; then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at  $180^\circ\text{C}$ . Trap temperatures up to  $220^\circ\text{C}$  may be employed; however, the higher temperature will shorten the useful life of the trap. After approximately 7 minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.4.1.14 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank organic-free reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.4.1.15 For matrix spike analysis, add 10  $\mu\text{L}$  of the matrix spike solution (Section 5.9) to the 5 mL of sample to be purged. Disregarding any dilutions, this is equivalent to a concentration of 50  $\mu\text{g/L}$  of each matrix spike standard.

7.4.1.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve. Proceed to Sections 7.5.1 and 7.5.2 for qualitative and quantitative analysis.

7.4.2 Compositing aqueous samples prior to GC/MS analysis.

7.4.2.1 Add 5 mL or equal larger amounts of each sample (up to 5 samples are allowed) to a 25 mL glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

7.4.2.2 The samples must be cooled at 4°C during this step to minimize volatilization losses.

7.4.2.3 Mix well and draw out a 5 mL aliquot for analysis.

7.4.2.4 Follow the sample analysis results above.

7.4.2.5 If less than 5 samples are used for compositing, a proportionately smaller syringe should be used unless a 25 mL sample is to be purged.

7.4.3 Water-miscible liquids

7.4.3.1 Water-miscible liquids are analyzed as water samples after first diluting them at least 50-fold with organic-free reagent water.

7.4.3.2 Initial and serial dilutions can be prepared by pipetting 2 mL of the sample to a 100 mL volumetric flask and diluting to volume with organic-free reagent water. Transfer immediately to a 5 mL gas tight syringe.

7.4.3.3 Alternatively, prepare dilutions directly in a 5 mL syringe filled with organic-free reagent water by adding at least 20  $\mu\text{L}$ , but not more than 100  $\mu\text{L}$  of liquid sample. The sample is ready for addition of internal and surrogate standards.

7.4.4 Sediment/soil and waste samples - It is highly recommended that all samples of this type be screened prior to the purge-and-trap GC/MS analysis by the headspace GC/FID method. These samples may contain percent quantities of purgeable organics that will contaminate the purge-and-trap system, and require extensive cleanup and instrument downtime. Use the screening data to determine whether to use the low-concentration method (0.005-1 mg/Kg) or the high-concentration method (> 1 mg/Kg).

7.4.4.1 Low-concentration method - This is designed for samples containing individual purgeable compounds of < 1 mg/Kg. It is limited to sediment/ soil samples and waste that is of a similar consistency (granular and porous). The low-concentration method is based on purging a heated sediment/soil sample mixed with organic-free reagent water containing the surrogate and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples.

7.4.4.1.1 Use a 5 g sample if the expected concentration is < 0.1 mg/Kg or a 1 g sample for expected concentrations between 0.1 and 1 mg/Kg.

7.4.4.1.2 The GC/MS system should be set up as in Sections 7.2 and 7.3. This should be done prior to the preparation of the sample to avoid loss of volatiles from standards and samples. A heated purge calibration curve must be prepared and used for the quantitation of all samples analyzed with the low-concentration method. Follow the initial and daily calibration instructions, except for the addition of a 40°C purge temperature.

7.4.4.1.3 Remove the plunger from a 5 mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 5.0 mL. Add 10 µL each of surrogate spiking solution (Section 5.5) and internal standard solution (Section 5.6) to the syringe through the valve (Surrogate spiking solution and internal standard solution may be mixed together.). The addition of 10 µL of the surrogate spiking solution to 5 g of sediment/soil is equivalent to 50 µg/Kg of each surrogate standard.

7.4.4.1.4 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh the amount determined in Section 7.4.4.1.1 into a tared purge device. Note and record the actual weight to the nearest 0.1 g.

7.4.4.1.5 Determination of sample % dry weight - In certain cases, sample results are desired based on dry weight basis. Percent dry weight may be determined by the following formula:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

7.4.4.1.6 Add the spiked water to the purge device, which contains the weighed amount of sample, and connect the device to the purge-and-trap system.

7.4.4.1.7 Heat the sample to 40 ± 1°C and purge the sample for 10.0 ± 0.1 minute.

7.4.4.1.8 Proceed with the analysis as outlined in Sections 7.4.1.11-7.4.1.16. Use 5 mL of the same organic-free reagent water as in the reagent blank. If saturated peaks occurred

or would occur if a 1 g sample were analyzed, the high-concentration method must be followed.

7.4.4.1.9 For low-concentration sediment/soils add 10  $\mu\text{L}$  of the matrix spike solution (Section 5.9) to the 5 mL of organic-free reagent water (Section 7.4.4.1.3). The concentration for a 5 g sample would be equivalent to 50  $\mu\text{g}/\text{Kg}$  of each matrix spike standard.

7.4.4.2 High-concentration method - The method is based on extracting the sediment/ soil with methanol. A waste sample is either extracted or diluted, depending on its solubility in methanol. An aliquot of the extract is added to organic-free reagent water containing surrogates, internal standards and, if applicable, matrix spike compounds. This is purged at ambient temperature. All samples with an expected concentration of  $> 1.0 \text{ mg}/\text{Kg}$  should be analyzed by this method.

7.4.4.2.1 The sample (for volatile organics) consists of the entire contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. For sediment/ soil and solid wastes that are insoluble in methanol, weigh 4 g (wet weight) of sample into a tared 20 mL vial. Use a top-loading balance. Note and record the actual weight to 0.1 gram and, if required, determine the percent dry weight of the sample using the procedure in Section 7.4.4.1.5.

7.4.4.2.2 For sediment/ soil or solid waste, quickly add 9.0 mL of appropriate solvent; then add 1.0 mL of the surrogate spiking solution to the vial. For a solvent miscible sample, dilute the sample to 10 mL with the appropriate solvent after adding 1.0 mL of the surrogate spiking solution. Cap and shake for 2 minutes.

[NOTE: Sections 7.4.4.2.1 and 7.4.4.2.2 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.]

7.4.4.2.3 Pipet approximately 1 mL of the extract to a GC vial for storage, using a disposable pipet. The remainder may be disposed of properly. Transfer approximately 1 mL of appropriate solvent to a separate GC vial for use as the method blank for each set of samples. These extracts may be stored at  $4^{\circ}\text{C}$  in the dark, prior to analysis.

7.4.4.2.4 The GC/MS system should be set up as in Sections 7.2 and 7.3. This should be done prior to the addition of the solvent extract to organic-free reagent water.

7.4.4.2.5 Table 10 can be used to determine the volume of solvent extract to add to the 5 mL of organic-free reagent water for analysis. If a screening procedure was followed (Method 3810 or 3820), use the estimated concentration to determine the appropriate volume. Otherwise, estimate the concentration range of the sample from the low-concentration analysis to determine the appropriate volume. If the sample was submitted

as a high-concentration sample, start with 100  $\mu$ L. All dilutions must keep the response of the major constituents (previously saturated peaks) in the upper half of the linear range of the curve.

7.4.4.2.6 Remove the plunger from a 5.0 mL Luer-lock type syringe equipped with a syringe valve and fill until overflowing with water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.9 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of the sample extract and of standards. Add 10  $\mu$ L of internal standard solution. Also add the volume of solvent extract determined in Section 7.4.4.2.5 and a volume of extraction or dissolution solvent to total 100  $\mu$ L (excluding methanol in standards).

7.4.4.2.7 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valve and inject the organic-free reagent water/methanol sample into the purging chamber.

7.4.4.2.8 Proceed with the analysis as outlined in Section 7.4.1.11-7.4.1.16. Analyze all reagent blanks on the same instrument as that used for the samples. The standards and blanks should also contain 100  $\mu$ L of solvent to simulate the sample conditions.

7.4.4.2.9 For a matrix spike in the high-concentration sediment/soil samples, add 8.0 mL of methanol, 1.0 mL of surrogate spike solution (Section 5.5), and 1.0 mL of matrix spike solution (Section 5.9) as in Section 7.4.4.2.2. This results in a 6,200  $\mu$ g/Kg concentration of each matrix spike standard when added to a 4 g sample. Add a 100  $\mu$ L aliquot of this extract to 5 mL of organic-free reagent water for purging (as per Section 7.4.4.2.6).

## 7.5 Data Interpretation

### 7.5.1 Qualitative Analysis

7.5.1.1 The qualitative identification of compounds determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds should be identified as present when the criteria below are met.

7.5.1.1.1 The RRT of the sample component is within  $\pm 0.06$  RRT units of the RRT of the standard component.

7.5.1.1.2 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.5.1.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.5.1.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

7.5.1.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When chromatographic peaks obviously represent more than one sample component (e.g., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (e.g., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

7.5.1.2 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the type of analyses being conducted. Guidelines for making tentative identification are:

- (1) Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.
- (2) The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
- (3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
- (4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
- (5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of

background contamination or coeluting peaks. Data system library reduction programs can sometimes create these discrepancies.

Computer generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual comparison of sample with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

### 7.5.2 Quantitative Analysis

7.5.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given analyte (See Table 6).

7.5.2.2 When MS response is linear and passes through the origin, calculate the concentration of each identified analyte in the sample as follows:

Water and Water-Miscible Waste:

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_{(x)})(I_{(s)})}{(A_{(is)})(\text{RF})(V_{(o)})}$$

where:

$A_{(x)}$  = Area of characteristic ion for compound being measured.

$I_{(s)}$  = Amount of internal standard injected (ng).

$A_{(is)}$  = Area of characteristic ion for the internal standard.

RF = Mean relative response factor for compound being measured (Section 7.2.7).

$V_{(o)}$  = Volume of water purged (mL), taking into consideration any dilutions made.

Sediment/ Soil, Sludge, and Waste:

High-concentration:

$$\text{Concentration } (\mu\text{g/Kg}) = \frac{(A_{(x)})(I_{(s)}) (V_{(i)})}{(A_{(is)})(\text{RF})(V_{(i)})(W_{(s)})}$$

Low-concentration:

$$\text{Concentration } (\mu\text{g/Kg}) = \frac{(A_{(x)})(I_{(s)})}{(A_{(is)})(\text{RF})(W_{(s)})}$$

where.

$A_{(x)}$   $I_{(s)}$   $A_{(is)}$  RF = Same as in water and water-miscible waste above.

$V_{(t)}$  = Volume of total extract ( $\mu\text{L}$ ) (use 10,000  $\mu\text{L}$  or a factor of this when dilutions are made).

$V_{(i)}$  = Volume of extract added ( $\mu\text{L}$ ) for purging.

$W_{(s)}$  = Weight of sample extracted or purged (g). The wet weight or dry weight may be used, depending upon the specific applications of the data.

7.5.2.4 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulae given above should be used with the following modifications: The areas  $A_{(x)}$  and  $A_{(is)}$  should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate ("J") and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

## 8.0 QUALITY CONTROL

8.1 Each laboratory that uses these methods is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document quality data. The laboratory should maintain records to document the quality of data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check sample should be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.2 Before processing any samples, the analyst should demonstrate, through the analysis of a calibration blank, that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is extracted or there is a change in reagents, a reagent blank should be processed as a safeguard against chronic laboratory contamination. The blank sample should be carried through all stages of sample preparation and measurement.

8.3 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the daily calibration standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked here are: Do the peaks look normal?; Is the response obtained

comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still useable, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system should take place.

#### 8.4 Required instrument QC

8.4.1 The GC/MS system must be tuned to meet the BFB specifications in Table 4.

8.4.2 There must be an initial calibration of the GC/MS system as specified in Section 7.2.

8.4.3 The GC/MS system must meet the SPCC criteria specified in Section 7.3.3 and the CCC criteria in Section 7.3.4, each 12 hours.

8.5 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.5.1 A quality control (QC) reference sample concentrate is required containing each analyte at a concentration of 10 mg/L in methanol. The QC reference sample concentrate may be prepared from pure standard materials or purchased as certified solutions. If prepared by the laboratory, the QC reference sample concentrate must be made using stock standards prepared independently from those used for calibration.

8.5.2 Prepare a QC reference sample to contain 20 µg/L of each analyte by adding 200 µL of QC reference sample concentrate to 100 mL of organic-free reagent water. For low level 25 mL sample, spike at 5 µg/L.

8.5.3 Four 5 mL aliquots (or 25 mL for low level) of the well-mixed QC reference sample are analyzed according to the method beginning in Section 7.4.1.

8.5.4 Calculate the average recovery ( $\bar{x}$ ) and the standard deviation of the recovery ( $s$ ), for the results.

8.5.5 Tables 7 and 8 provide single laboratory recovery and precision data obtained for the method analytes from water. Similar results from dosed water should be expected by any experienced laboratory. Compare results obtained in Step 8.5.4 to the single laboratory recovery and precision data. If the results are not comparable, review potential problem area and repeat the test. Results are comparable if the calculated standard deviation of the recovery does not exceed 2.6 times the single laboratory RSD or 20%, whichever is greater, and the mean recovery lies within the interval  $\bar{x} \pm 3s$  or  $\bar{x} \pm 30\%$ , whichever is greater.

8.5.6 When one or more of the analytes tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.5.6.1 or 8.5.6.2.

8.5.6.1 Locate and correct the source of the problem and repeat the test for all analytes beginning with Section 8.5.2.

8.5.6.2 Beginning with Section 8.5.2, repeat the test only for those analytes that are not comparable. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.5.2.

8.6 For each analytical batch (up to 20 samples), a reagent blank, a matrix spike, duplicate or matrix spike duplicate, and a blank spike (LCS) should be analyzed (the frequency of the spikes may be different for different programs). The blank and spiked samples should be carried through all stages of the sample preparation and measurement steps.

8.6.1 The concentration of the spike in the sample should be determined as follows:

8.6.1.1 If, as in compliance monitoring, the concentration of a specific analyte in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Step 8.6.2, whichever concentration would be larger.

8.6.1.2 If the concentration of a specific analyte in a water sample is not being checked against a specific limit, the spike should be at 20 µg/L (or 5 µg/L for low level) or 1 to 5 times higher than the background concentration determined in Section 8.6.2, whichever concentration would be larger. For other matrices, the recommended spiking concentration is 10 times the EQL.

8.6.2 Analyze one 5 mL sample aliquot (or 25 mL for low level) to determine the background concentration (B) of each analyte. If necessary, prepare a new QC reference sample concentrate (Section 8.5.1) appropriate for the background concentration in the sample. Spike a second 5 mL (or 25 mL for low level) sample aliquot with 10 µL of the QC reference sample concentrate and analyze it to determine the concentration after spiking (A) of each analyte. Calculate each percent recovery (p) as  $100(A-B)/T$ , where T is the known true value of the spike.

8.6.2.1 Compare the percent recovery (R<sub>i</sub>) for each analyte with QC acceptance criteria established from the analyses of laboratory control standards (Section 8.5). Monitor all data from dosed samples.

8.6.2.2 If recovery is not within limits, the following procedures are required.

8.6.2.2.1 Check to be sure there are no errors in calculations, matrix spike solution and internal standards. Also, check instrument performance.

8.6.2.2.2 Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.

8.6.2.2.3 If the checks in 8.6.2.2.1 reveal no errors, the recovery problem encountered with the dosed sample is judged to be matrix-related, not system-related. The result for that analyte in the unspiked sample is labeled to inform the user that the results are suspect due to matrix effects.

8.7 As part of the QC program for the laboratory, method accuracy for each matrix studied should be assessed and records should be maintained. After the analysis of five spiked samples (of the same matrix) as in Step 8.6, calculate the average percent recovery (the average of  $p$ ) and the standard deviation of the percent recovery ( $sp$ ). Express the accuracy assessment as a percent recovery interval from the average of  $p - 2sp$ . If the average of  $p = 90\%$  and  $sp = 10\%$ , for example, the accuracy interval is expressed as 70-100%. Update the accuracy assessment for each analyte on a regular basis.

8.8 To determine acceptable accuracy and precision limits for surrogate standards the following procedure should be performed.

8.8.1 For each sample analyzed, calculate the percent recovery of each surrogate in the sample.

8.8.2 Once a minimum of thirty samples of the same matrix have been analyzed, calculate the average percent recovery (the average of  $p$ ) and standard deviation of the percent recovery ( $sp$ ) for each of the surrogates.

8.8.3 For a given matrix, calculate the upper and lower control limit for method performance for each surrogate standard. This should be done as follows:

$$\text{Upper Control Limit(UCL)} = p + 3sp$$

$$\text{Lower Control Limit(LCL)} = p - 3sp$$

8.8.4 For aqueous and soil matrices, laboratory established surrogate control limits should, if applicable, be compared with the control limits listed in Table 9. The limits given in Table 9 are multi-laboratory performance based limits for soil and aqueous samples, and therefore, the single-laboratory limits established in Section 8.8.3 should fall within those given in Table 9 for these matrices.

8.8.5 If recovery is not within limits, the following procedures are required.

\* Check to be sure there are no errors, in calculations, surrogates solution and internal standards. Also, check instrument performance.

- \* Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- \* Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration".

8.8.6 At a minimum, each laboratory should update surrogate recovery limits on a matrix-by-matrix basis, annually.

8.9 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column or a different ionization mode using a mass spectrometer should be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.10 In recognition of the rapid advances occurring chromatography, the analyst is permitted to modify GC columns, GC conditions, or detectors to improve the separations or lower the cost of the measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Section 8.4 and to document any such changes in the SOP.

## 9.0 CORRECTIVE ACTION

Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method Detection Limit (MDL) Study	Annually for water and soil.	Minimum of 7 replicates. Spike level must be at a concentration between the calculated MDL and 10 x MDL.	Reprepare study at adjusted spiking levels for MDLs falling outside of acceptable range.
Initial Demonstration of Proficiency	Prior to use of method for client samples.	See Table 7 or 8.	Investigate failure. Verify solution integrity and instrument performance. Reprepate and reanalyze.

Tuning	Every 12 hr, prior to analysis.	See Table 4.	Inspect instrument and perform necessary maintenance. Retune instrument and reanalyze any samples since last acceptable tune.
Initial Calibration	As necessary.	Minimum of five points. Low standard must not exceed method reporting limit. Minimum RRF for each SPCC and maximum %RSD for each CCC. See Section 7.2 of this SOP.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
Secondary Source Calibration Verification	Following every initial calibration.	Secondary source check - all target compounds +/- 20% of expected value.	Verify solution integrity and instrument performance. Reanalyze, if still out, then recalibrate.
Continuing Calibration Verification	Every 12 hr.	All CCCs must be less than 20% different from expected value.	Verify solution integrity and instrument performance. Reanalyze standard once, if still out recalibrate and reanalyze affected samples.
Method Blanks	One every 12 hr.	Target compounds < reporting limit.	Investigate source of contamination. Reprepare and reanalyze all associated samples.
Surrogate Spike	Every sample, blank and standard.	See Table 9 or in-house generated limits.	Check calculations. If still out, verify instrument performance and correct, if necessary. Reanalyze sample, if still out reextract and reanalyze, report both sets of data.

Laboratory Control Sample	One per analytical batch.	Control limits generated from in-house data.	Reanalyze once, if still out verify solution integrity and instrument performance. Reprepare and reanalyze all associated samples.
Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per 20 samples of a similar matrix.	Control limits generated from in-house data.	Reanalyze once, if still out verify solution integrity and instrument performance. If LCS acceptable, narrate as possible matrix effect.

## 10.0 METHOD PERFORMANCE

10.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

10.2 This method has been tested in a single laboratory using spiked water. Using a wide-bore capillary column, water was spiked at concentrations between 0.5 and 10 ug/L. Single laboratory accuracy and precision data are presented for the method analytes in Table 7. Calculated MDLs are presented in Table 1.

10.3 The method was tested using water spiked at 0.1 to 0.5 ug/L and analyzed on a cryofocussed narrow-bore column. The accuracy and precision data for these compounds are presented in Table 8. MDL values were also calculated from these data and are presented in Table 2.

## 11.0 REFERENCES

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**TABLE 1. CHROMATOGRAPHIC RETENTION TIMES AND METHOD  
 DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON  
 WIDE BORE CAPILLARY COLUMNS**

Analyte	Retention Time (min)			MDL (d) (ug/L)
	Column 1(a)	Column 2(b)	Column 2(c)	
Dichlorodifluoromethane	1.55	0.70	3.13	0.10
Chloromethane	1.63	0.73	3.40	0.13
Vinyl Chloride	1.71	0.79	3.93	0.17
Bromomethane	2.01	0.96	4.80	0.11
Chloroethane	2.09	1.02	--	0.10
Trichlorofluoromethane	2.27	1.19	6.20	0.08
1,1-Dichloroethene	2.89	1.57	7.83	0.12
Methylene chloride	3.60	2.06	9.27	0.03
trans-1,2-Dichloroethene	3.98	2.36	9.90	0.06
1,1-Dichloroethane	4.85	2.93	10.80	0.04
2,2-Dichloropropane	6.01	3.80	11.87	0.35
cis-1,2-Dichloroethene	6.19	3.90	11.93	0.12
Chloroform	6.40	4.80	12.60	0.03
Bromochloromethane	6.74	4.38	12.37	0.04
1,1,1-Trichloroethane	7.27	4.84	12.83	0.08
Carbon tetrachloride	7.61	5.26	13.17	0.21
1,1-Dichloropropene	7.68	5.29	13.10	0.10
Benzene	8.23	5.67	13.50	0.04
1,2-Dichloroethane	8.40	5.83	13.63	0.06
Trichloroethene	9.59	7.27	14.80	0.19
1,2-Dichloropropane	10.09	7.66	15.20	0.04
Bromodichloromethane	10.59	8.49	15.80	0.08
Dibromomethane	10.65	7.93	15.43	0.24
trans-1,3-Dichloropropene	--	--	16.70	--
Toluene	12.43	10.00	17.40	0.11
cis-1,3-Dichloropropene	--	--	17.90	--
1,1,2-Trichloroethane	13.41	11.05	18.30	0.10
Tetrachloroethene	13.74	11.15	18.60	0.14
1,3-Dichloropropane	14.04	11.31	18.70	0.04
Dibromochloromethane	14.39	11.85	19.20	0.05
1,2-Dibromoethane	14.73	11.83	19.40	0.06
1-Chlorohexane	15.46	13.29	--	0.05
Chlorobenzene	15.76	13.01	20.67	0.04
1,1,1,2-Tetrachloroethane	15.94	13.33	20.87	0.05
Ethylbenzene	15.99	13.39	21.00	0.06

p-Xylene	16.12	13.69	21.30	0.13
m-Xylene	16.17	13.68	21.37	0.05
o-Xylene	17.11	14.52	22.27	0.11
Styrene	17.31	14.60	22.40	0.04
Bromoform	17.93	14.88	22.77	0.12
Isopropylbenzene	18.06	15.46	23.30	0.15
1,1,2,2-Tetrachloroethane	18.72	16.35	24.07	0.04
Bromobenzene	18.95	15.86	24.00	0.03
1,2,3-Trichloropropane	19.02	16.23	24.13	0.32
n-Propylbenzene	19.06	16.41	24.33	0.04
2-Chlorotoluene	19.34	16.42	24.53	0.04
1,3,5-Trimethylbenzene	19.47	16.90	24.83	0.05
4-Chlorotoluene	19.50	16.72	24.77	0.06
tert-Butylbenzene	20.28	17.57	26.60	0.14
1,2,4-Trimethylbenzene	20.34	17.70	31.50	0.13
sec-Butylbenzene	20.79	18.09	26.13	0.13
p-Isopropyltoluene	21.20	18.52	26.50	0.12
1,3-Dichlorobenzene	21.22	18.14	26.37	0.12
1,4-Dichlorobenzene	21.55	18.39	26.60	0.03
n-Butylbenzene	22.22	19.49	27.32	0.11
1,2-Dichlorobenzene	22.52	19.17	27.43	0.03
1,2-Dibromo-3-chloropropane	24.53	21.08	--	0.26
1,2,4-Trichlorobenzene	26.55	23.08	31.50	0.04
Hexachlorobutadiene	26.99	23.68	32.07	0.11
Naphthalene	27.17	23.52	32.20	0.04
1,2,3-Trichlorobenzene	27.78	24.18	32.97	0.03
4-Bromofluorobenzene (Surr)	18.63	15.71	23.63	--

(a) = Column 1 - 60 meter x 0.75 mm ID VOCOL capillary. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

(b) = Column 2 - 30 meter x 0.53 mm ID DB-624 wide-bore capillary using cryogenic oven. Hold at 10°C for 5 minutes, then program to 160°C at 6°C/min.

(c) = Column 2' - 30 meter x 0.53 mm ID DB-624 wide-bore capillary, cooling GC oven to ambient temperatures. Hold at 10°C for 6 minutes, program to 70°C at 10°C/min, program to 120°C at 5°C/min, then program to 180°C at 8°C/min.

(d) = MDL based on a 25 mL sample volume.

**TABLE 2. CHROMATOGRAPHIC RETENTION TIMES AND METHOD  
 DETECTION LIMITS (MDL) FOR VOLATILE ORGANIC COMPOUNDS ON  
 NARROW BORE CAPILLARY COLUMNS**

Analyte	Retention Time (min) Column 3(a)	MDL (d) ug/L
Dichlorodifluoromethane	0.88	0.11
Chloromethane	0.97	0.05
Vinyl chloride	1.04	0.04
Bromomethane	1.29	0.06
Chloroethane	1.45	0.02
Trichlorofluoromethane	1.77	0.07
1,1-Dichloroethene	2.33	0.05
Methylene chloride	2.66	0.09
trans-1,2-Dichloroethene	3.54	0.03
1,1-Dichloroethane	4.03	0.03
cis-1,2-Dichloroethene	5.07	0.06
2,2-Dichloropropane	5.31	0.08
Chloroform	5.55	0.04
Bromochloromethane	5.63	0.09
1,1,1-Trichloroethane	6.76	0.04
1,2-Dichloroethane	7.00	0.02
1,1-Dichloropropene	7.16	0.12
Carbon tetrachloride	7.41	0.02
Benzene	7.41	0.03
1,2-Dichloropropane	8.94	0.02
Trichloroethene	9.02	0.02
Dibromomethane	9.09	0.01
Bromodichloromethane	9.34	0.03
Toluene	11.51	0.08
1,1,2-Trichloroethane	11.99	0.08
1,3-Dichloropropane	12.48	0.08
Dibromochloromethane	12.80	0.07
Tetrachloroethene	13.20	0.05
1,2-Dibromoethane	13.60	0.10
Chlorobenzene	14.33	0.03
1,1,1,2-Tetrachloroethane	14.73	0.07
Ethylbenzene	14.73	0.03
p-Xylene	15.30	0.06
m-Xylene	15.30	0.03
Bromoform	15.70	0.20
o-Xylene	15.78	0.06
Styrene	15.78	0.27
1,1,2,2-Tetrachloroethane	15.78	0.20

1,2,3-Trichloropropane	16.26	0.09
Isopropylbenzene	16.42	0.10
Bromobenzene	16.42	0.11
2-Chlorotoluene	16.74	0.08
n-Propylbenzene	16.82	0.10
4-Chlorotoluene	16.82	0.06
1,3,5-Trimethylbenzene	16.99	0.06
tert-Butylbenzene	17.31	0.33
1,2,4-Trimethylbenzene	17.31	0.09
sec-Butylbenzene	17.47	0.12
1,3-Dichlorobenzene	17.47	0.05
p-Isopropyltoluene	17.63	0.26
1,4-Dichlorobenzene	17.63	0.04
1,2-Dichlorobenzene	17.79	0.05
n-Butylbenzene	17.95	0.10
1,2-Dibromo-3-chloropropane	18.03	0.50
1,2,4-Trichlorobenzene	18.84	0.20
Naphthalene	19.07	0.10
Hexachlorobutadiene	19.24	0.10
1,2,3-Trichlorobenzene	19.24	0.14

(a) = Column 3 - 30 meter x 0.32 mm ID DB-5 capillary with 1 um film thickness.

(b) = MDL based on a 25 mL sample volume.

**TABLE 3. ESTIMATED QUANTITATION LIMITS FOR VOLATILE ANALYTES(a)**

	Groundwater ug/L	Low Soil/Sediment(b) ug/Kg
5 mL of Water Purged	5	
25 mL of Water Purged	1	
Soil/ Sediment		5

(a) = Estimated Quantitation Limit (EQL) - The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The EQL is generally 5 to 10 times the MDL. However, it may be nominally chosen within these guidelines to simplify data reporting. For many analytes the EQL analyte concentration is selected for the lowest non-zero standard in the calibration curve. Sample EQLs are highly matrix-dependent. The EQLs listed herein are provided for guidance and may not always be achievable. See the following information for further guidance on matrix-dependent EQLs.

(b) = EQLs listed for soil/sediment are based on wet weight. Normally data is reported on a dry weight basis; therefore, EQLs will be higher, based on the percent dry weight in each sample.

Other Matrices	Factor(c)
Water miscible liquid waste	50
High-concentration soil and sludge	125
Non-water miscible waste	500

(c)EQL = [EQL for low soil sediment (Table 3)] X [Factor]. For non-aqueous samples, the factor is on a wet-weight basis.

**TABLE 4. BFB MASS - INTENSITY SPECIFICATIONS (4-BROMO-FLUOROBENZENE)**

Mass	Intensity Required (relative abundance)
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

**TABLE 5. CHARACTERISTIC MASSES (M/Z) FOR PURGEABLE ORGANIC COMPOUNDS**

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Acetone	43	58
Benzene	78	--
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91, 134
Carbon disulfide	76	78
Carbon tetrachloride	117	119
Chlorobenzene	112	77, 114
Chlorodibromomethane	129	127
Chloroethane	64	66
Chloroform	83	85
1, Chlorohexane	91	--
Chloromethane	50	52
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,2-Dibromo-3-chloropropane	157	75, 155
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
cis-1,2-Dichloroethene	96	61, 98
trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
cis-1,3-Dichloropropene	75	77, 39
trans-1,3-Dichloropropene	75	77, 39

Ethylbenzene	106	91
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Isopropylbenzene	105	120
p-Isopropyltoluene	119	134, 91
Methylene chloride	84	86, 49
4-Methyl-2-pentanone	43	100, 58, 85
Naphthalene	128	--
n-Propylbenzene	91	120
Styrene	104	78
1,1,1,2-Tetrachloroethane	133	131, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	131, 166, 168, 129
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	130	95, 132
Trichlorofluoromethane	101	103
1,2,3-Trichloropropane	75	77
Trichlorotrifluoroethane	151	--
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
<b>INTERNAL</b>	<b>Primary</b>	<b>Secondary</b>
<b>STANDARDS/SURROGATES</b>	<b>Characteristic Ion</b>	<b>Characteristic Ion(s)</b>
4-Bromofluorobenzene (Surr)	95	174, 176
Dibromofluoromethane (Surr)	113	
Toluene-d(8) (Surr)	98	
Pentafluorobenzene (IS)	168	
1,4-Difluorobenzene (IS)	114	
Chlorobenzene-d(5) (IS)	117	
1,2-Dichloroethane-d(4) (Surr)	65	
1,4-Dichlorobenzene-d(4) (IS)	152	115, 150

**TABLE 6. VOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION**

Pentafluorobenzene	1,4-Difluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Acetone	Benzene	Bromofluorobenzene (Surr)	Bromobenzene
Bromochloromethane	Bromodichloromethane	1,2-Dibromoethane	n-Butylbenzene
Carbon tetrachloride	Dibromomethane	1-Chlorohexane	sec-Butylbenzene
Bromomethane	1,2-Dichloroethane	trans-1,3-Dichloropropene	tert-Butylbenzene
2-Butanone	1,2-Dichloropropane	Toluene	2-Chlorotoluene
Carbon disulfide	cis-1,3-Dichloropropene	Toluene-d(8)(Surr)	4-Chlorotoluene
Chloroethane	4-Methyl-2-pentanone	1,1,2-Trichloroethane	1,2-Dibromo-3-chloropropane
Chloroform	Trichloroethene	m/p-Xylene	1,2-Dichlorobenzene
1,2-Dichloroethane-d(4)(Surr)		o-Xylene	1,3-Dichlorobenzene
Chloromethane		Bromoform	1,4-Dichlorobenzene
Dibromofluoromethane (Surr.)		Chlorodibromomethane	Hexachlorobutadiene
Dichlorodifluoromethane		Chlorobenzene	p-Isopropyltoluene
1,1-Dichloropropene		1,3-Dichloropropane	Naphthalene
1,1-Dichloroethane		Ethylbenzene	n-Propylbenzene
1,1-Dichloroethene		2-Hexanone	1,1,2,2-Tetrachloroethane
cis-1,2-Dichloroethene		Styrene	1,2,3-Trichlorobenzene
trans-1,2-Dichloroethene		1,1,1,2-Tetrachloroethane	1,2,4-Trichlorobenzene
2,2-Dichloropropane		Isopropyl benzene	1,2,3-Trichloropropane
Iodomethane		Tetrachloroethene	1,2,4-Trimethylbenzene
Methylene chloride		Xylene	1,3,5-Trimethylbenzene
1,1,1-Trichloroethane			
Trichlorofluoromethane			
Trichlorotri-fluoroethane			
Vinyl acetate			
Vinyl chloride			

**TABLE 7. SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A WIDE BORE CAPILLARY COLUMN**

Analyte	Conc. Range ug/L	No. of Samples	% Rec	Std Dev of Recovery(b)	%RSD
Benzene	0.1 - 10	31	97	6.5	5.7
Bromobenzene	0.1 - 10	30	100	5.5	5.5
Bromochloromethane	0.5 - 10	24	90	5.7	6.4
Bromodichloromethane	0.1 - 10	30	95	5.7	6.1
Bromoform	0.5 - 10	18	101	6.4	6.3
Bromomethane	0.5 - 10	18	95	7.8	8.2
n-Butylbenzene	0.5 - 10	18	100	7.6	7.6
sec-Butylbenzene	0.5 - 10	16	100	7.6	7.6
tert-Butylbenzene	0.5 - 10	18	102	7.4	7.3
Carbon tetrachloride	0.5 - 10	24	84	7.4	8.8
Chlorobenzene	0.1 - 10	31	98	5.8	5.9
Chloroethane	0.5 - 10	24	89	8.0	9.0
Chloroform	0.5 - 10	24	90	5.5	6.1
Chloromethane	0.5 - 10	23	93	8.3	8.9
2-Chlorotoluene	0.1 - 10	31	90	5.6	6.2
4-Chlorotoluene	0.1 - 10	31	98	8.2	8.3
1,2-Dibromo-3-Chloropropane	0.5 - 10	24	83	16.6	19.9
Dibromochloromethane	0.1 - 10	31	92	6.5	7.0
1,2-Dibromoethane	0.5 - 10	24	102	4.0	3.9
Dibromomethane	0.5 - 10	24	100	5.6	5.6
1,2-Dichlorobenzene	0.1 - 10	31	93	5.8	6.2
1,3-Dichlorobenzene	0.5 - 10	24	99	6.8	6.9
1,4-Dichlorobenzene	0.2 - 20	31	103	6.6	6.4
Dichlorodifluoromethane	0.5 - 10	18	90	6.9	7.7
1,1-Dichlorobenzene	0.5 - 10	24	96	5.1	5.3
1,2-Dichlorobenzene	0.1 - 10	31	95	5.1	5.4
1,1-Dichloroethene	0.1 - 10	34	94	6.3	6.7
cis-1,2-Dichloroethene	0.5 - 10	18	101	6.7	6.7
trans-1,2-Dichloroethene	0.1 - 10	30	93	5.2	5.6
1,2-Dichloropropane	0.1 - 10	30	97	5.6	6.1
1,3-Dichloropropane	0.1 - 10	31	96	5.7	6.0
2,2-Dichloropropane	0.5 - 10	12	86	14.6	16.9
1,1-Dichloropropene	0.5 - 10	18	98	8.7	8.9
Ethylbenzene	0.1 - 10	31	99	8.4	8.6
Hexachlorobutadiene	0.5 - 10	18	100	6.8	6.8
Isopropylbenzene	0.5 - 10	16	101	7.7	7.6

p-Isopropyltoluene	0.1 - 10	23	99	6.7	6.7
Methylene chloride	0.1 - 10	30	95	5.0	5.3
Naphthalene	0.1 -100	31	104	8.6	8.2
n-Propylbenzene	0.1 - 10	31	100	5.8	5.8
Styrene	0.1 -100	39	102	7.3	7.2
1,1,1,2-Tetrachloroethane	0.5 - 10	24	90	6.1	6.8
1,1,2,2-Tetrachloroethane	0.1 - 10	30	91	5.7	6.3
Tetrachloroethene	0.5 - 10	24	89	6.0	6.8
Toluene	0.5 - 10	18	102	8.	8.0
1,2,3-Trichlorobenzene	0.5 - 10	18	109	9.4	8.6
1,2,4-Trichlorobenzene	0.5 - 10	18	108	9.0	8.3
1,1,1-Trichloroethane	0.5 - 10	18	98	7.9	8.1
1,1,2-Trichloroethane	0.5 - 10	18	104	7.6	7.3
Trichloroethene	0.5 - 10	24	90	6.5	7.3
Trichlorofluoromethane	0.5 - 10	24	89	7.2	8.1
1,2,3-Trichloropropane	0.5 - 10	16	108	15.6	14.4
1,2,4-Trimethylbenzene	0.5 - 10	18	99	8.0	8.1
1,3,5-Trimethylbenzene	0.5 - 10	23	92	6.8	7.4
Vinyl chloride	0.5 - 10	18	98	6.5	6.7
o-Xylene	0.1 - 31	18	103	7.4	7.2
m-Xylene	0.1 - 10	31	97	6.3	6.5
p-Xylene	0.5 - 10	18	104	8.0	7.7

(a) = Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

(b) = Standard deviation was calculated by pooling data form three concentrations.

**TABLE 8. SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR VOLATILE ORGANIC COMPOUNDS IN WATER DETERMINED WITH A NARROW BORE CAPILLARY COLUMN**

Analyte	Conc. ug/L	Number of Samples	%Reco- very (a)	Std. Dev. of Recovery	%RSD
Benzene	0.1	7	99	6.2	6.3
Bromobenzene	0.5	7	97	7.4	7.6
Bromochloromethane	0.5	7	97	5.8	6.0
Bromodichloromethane	0.1	7	100	4.6	4.6
Bromoform	0.5	7	101	5.4	5.3
Bromomethane	0.5	7	99	7.1	7.2
n-Butylbenzene	0.5	7	94	6.0	6.4
sec-Butylbenzene	0.5	7	110	7.1	6.5
tert-Butylbenzene	0.5	7	110	2.5	2.3
Carbon tetrachloride	0.1	7	108	6.8	6.3
Chlorobenzene	0.1	7	91	5.8	6.4
Chloroethane	0.1	7	100	5.8	5.8
Chloroform	0.1	7	105	3.2	3.0
Chloromethane	0.5	7	101	4.7	4.7
2-Chlorotoluene	0.5	7	99	4.6	4.6
4-Chlorotoluene	0.5	7	96	7.0	7.3
1,2-Dibromo-3-chloropropane	0.5	7	92	10.0	10.9
Dibromochloromethane	0.1	7	99	5.6	5.7
1,2-Dibromoethane	0.5	7	97	5.6	5.8
Dibromomethane	0.5	7	93	5.6	6.0
1,2-Dichlorobenzene	0.1	7	97	3.5	3.6
1,3-Dichlorobenzene	0.1	7	101	6.0	5.9
1,4-Dichlorobenzene	0.1	7	106	6.5	6.1
Dichlorodifluoromethane	0.1	7	99	8.8	8.9
1,1-Dichloroethane	0.5	7	98	6.2	6.3
1,2-Dichloroethane	0.1	7	100	6.3	6.3
1,1-Dichloroethene	0.1	7	95	9.0	9.5
cis-1,2-Dichloroethene	0.1	7	100	3.7	3.7
trans-1,2-Dichloroethene	0.1	7	98	7.2	7.3
1,2-Dichloropropane	0.5	7	96	6.0	6.3
1,3-Dichloropropane	0.5	7	99	4.9	4.9
1,1-Dichloropropene	0.5	7	102	7.4	7.3
Ethylbenzene	0.5	7	99	5.2	5.3
Hexachlorobutadiene	0.5	7	100	6.7	6.7
Isopropylbenzene	0.5	7	102	6.4	6.3
p-Isopropyltoluene	0.5	7	113	13.0	11.5
Methylene chloride	0.5	7	97	13.0	13.4

Naphthalene	0.5	7	98	7.2	7.3
n-Propylbenzene	0.5	7	99	6.6	6.7
Styrene	0.5	7	96	19.0	19.8
1,1,1,2-Tetrachloroethane	0.5	7	100	4.7	4.7
1,1,2,2-Tetrachloroethane	0.5	7	100	12.0	12.0
Tetrachloroethene	0.1	7	96	5.0	5.2
Toluene	0.5	7	100	5.9	5.9
1,2,3-Trichlorobenzene	0.5	7	102	8.9	8.7
1,2,4-Trichlorobenzene	0.5	7	91	16.0	17.6
1,1,1-Trichloroethane	0.5	7	100	4.0	4.0
1,1,2-Trichloroethane	0.5	7	102	4.9	4.8
Trichloroethene	0.1	7	104	2.0	1.9
Trichlorofluoromethane	0.1	7	97	4.6	4.7
1,2,3-Trichloropropane	0.5	7	96	6.5	6.8
1,2,4-Trimethylbenzene	0.5	7	96	6.5	6.8
1,3,5-Trimethylbenzene	0.5	7	101	4.2	4.2
Vinyl chloride	0.1	7	104	0.2	0.2
o-Xylene	0.5	7	106	7.5	7.1
m-Xylene	0.5	7	106	4.6	4.3
p-Xylene	0.5	7	97	6.1	6.3

(a) = Recoveries were calculated using internal standard method. Internal standard was fluorobenzene.

**TABLE 9. SURROGATE SPIKE RECOVERY LIMITS FOR WATER AND SOIL/SEDIMENT SAMPLES**

<b>Surrogate Compound</b>	<b>Low/High Water</b>	<b>Low/High Soil/Sediment</b>
4-Bromofluorobenzene(a)	86-115	74-121
Dibromofluoromethane(a)	86-118	80-120
Toluene-d8(a)	88-110	81-117
1,2-Dichloroethane-d4(a)	80-120	80-120

(a) = Single laboratory data for guidance only.

**TABLE 10. QUANTITY OF EXTRACT REQUIRED FOR ANALYSIS OF HIGH-CONCENTRATION SAMPLES**

<b>Approximate Concentration Range</b>	<b>Volume of Extract(a)</b>
500 - 10,000 ug/Kg	100 uL
1,000 - 20,000 ug/Kg	50 uL
5,000 - 100,000 ug/Kg	10 uL
25,000 - 500,000 ug/Kg	100 uL of 1/50 dilution(b)

Calculate appropriate dilution factor for concentrations exceeding this table.

(a) The volume of solvent added to 5 mL of water being purged should be kept constant. Therefore, add to the 5 mL syringe whatever volume of solvent is necessary to maintain a volume of 100 uL added to the syringe.

(b) Dilute an aliquot of the solvent extract and then take 100 uL for analysis.

**APPENDIX H**

**ANALYTICAL PROCEDURES FOR THE  
ORGANIC AND INORGANIC  
GEOCHEMISTRY LABS AT UW**

## Analytical Methods Used by the Organic Geochemistry Lab and the Inorganic Geochemistry Lab at UW

### 1: Anions by Anion Exchange Chromatography

Species Analysed: Acetate, bromide, chloride, nitrate, nitrite, phosphate, sulphate  
Analyses provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences  
Method provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences

The anions are analysed using a Dionex AS3 or AS4A anion exchange column. The utilization of a Dionex Micro Membrane Suppressor Column increases stability. The instrument used is either a Dionex System 2000 ion chromatograph or a Waters Ion Chromatograph utilizing a WISP 710B sampler, a model 745 Data Module, a model 510 pump and a model 430 Conductivity detector. The method of detection for both systems is conductivity.

An Alpkem Perstorp Analytical Environmental FLOW Solution system is also used to analyse some anions. This is a colorimetric system detecting the absorbance at specific wavelengths.

The samples are filtered (0.45 µm) and kept at 4°C until analysed. The instruments are operated using manufacturers' specifications. The results are reported as milligrams per litre.

A daily run of twenty to fifty samples contains 10 to 20 in-house standards. A setpoint standard is also run at least twice during the run in order to maintain in-house standard quality. The samples are rerun if the setpoint standard does not come within five percent of the stated value. Charge balances are done on all samples where possible. The Water Quality Lab has also participated in inter-laboratory comparisons in order to maintain in-house standard quality.

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### 2: Anions by Ion Chromatography, APHA 4110 (adapted)

#### 2.1 Method Summary

Species Analysed: Acetate, bromide, chloride, nitrate, nitrite, phosphate, sulphate

Analyses provided by: Accutest Laboratories Ltd., 146 Colonnade Road, Unit 8, Nepean, Ont., K2E 7Y1

Method provided by: Accutest Laboratories Ltd.

Sampling and Storage: Samples should be collected in a clean plastic bottle with a minimum volume of 100 mL

Equipment: Whatman #1 filters

Ion Chromatography Acrodisc 0.45 µm, 13 mm (Gelman #4485)

5-10 mL Luer Lock syringe

1 mL Luer syringe

Table 1: Sample schedule for anion analysis by ion chromatography.

Sample #	Name	Method	Datafile
1	standard	ANIONS.MET	ICyyddd
2	standard		
3	standard		
4	standard		
5	standard		
6	blank		
7	setpoint minerals		
8	setpoint PO <sub>4</sub> , NO <sub>3</sub>		
9	setpoint F		
10	blank		
11	standard		
12	standard		
13...22	samples		
23	duplicate		
24	standard		
25...34	samples		
35	duplicate		
36	setpoint or standard		
etc.	note: max = 99 but sampler will hold only 11 racks (66 samples)		
LAST	last method should be SHUT.MET turns off pump in IC		

5 mL Polyvials with filter caps (Dionex #038141)  
AG4A-SC guard column (4X50 mm)(Dionex #043175)  
AS4A-SC analytical column (4X250 mm)(Dionex #043174)  
ASRS-1 (4 mm)(Dionex #043189) anion suppressor  
Dionex ASM autosampler  
Dionex ACI Computer Interface  
Akran 433 486/33 computer system  
AI-450 Chromatography software (running under Windows 3.1)

**Reagents:**

Deionized distilled water > 16.7 Mohm  
 $\text{Na}_2\text{CO}_3$ ,  $\text{NaHCO}_3$ ,  $\text{NaCl}$ ,  $\text{NaBr}$ ,  $\text{NaF}$ ,  $\text{NaNO}_3$ ,  $\text{NaNO}_2$ ,  $\text{KH}_2\text{PO}_4$ ,  $\text{K}_2\text{SO}_4$

**2.2 Reagent Preparation**

1. Eluent concentrate: Dissolve 1.91 g of  $\text{Na}_2\text{CO}_3$  and 1.43 g of  $\text{NaHCO}_3$  in 100 mL of DDI water.
2. Eluent: Dilute 10 mL of concentrate to 1000 mL with DDI water. Prepare at least 2 litres at a time.

**2.3 Analysis**

**NOTE:** The following procedure assumes a working knowledge of Windows 3.1, Dionex AI-450 software, DX-100 Ion Chromatograph.

1. Prepare sample list and create an analysis schedule using the schedule program in the AI-450 software.
2. The schedule should be made up as follows:  
The standard at sample # 24 is run as an instrument condition check, if response time and values not within tolerance (10%) instrument must be recalibrated. 1 replicate and 1 standard for every 10 samples (= 2 sample racks). Matrix spikes may be added to list as necessary (5% of samples). Note: method is ANIONS.MET, datafile - ICyyddd = IC, year,day number; last sample name is SHUTOFF, datafile = lastone.
3. Use samples from unpreserved containers. If necessary filter through Ion Chromatography Acrodisc 0.45  $\mu\text{m}$ , 13 mm into 5 mL Polyvials in the same order as the schedule. Take care to put samples in proper order in the racks and to not mix up the racks. If samples are very turbid, filter through Whatrman #1.

4. Load racks into autosampler as they are filled to prevent errors.
5. Press run on the autosampler to advance to the first vial. Put the DX-100 into Relay control. Ensure that ACI is turned on.
6. Go to Run program in AI-450 software and load the schedule to be analysed.
7. Start the run.

#### 2.4 Calculation of Results

1. AI-450 program will calculate results based on calibration curve of each anion. The curves should be checked for linearity and order determined by shape of the curve. Ideally they should be linear ( $r^2 > 0.99$ ). Fit type is quadratic, better fits due to use of low end of instrument range (for this scale :  $30\mu\text{S}$ ).
2. If dilutions are entered into the schedule they will automatically be calculated.
3. Results should be reported to the precision required for each Anion.

#### 2.5 Quality Control

1. Quality control as per standard protocol: 10% duplicates, blanks and standards run daily.
2. Duplicates should be within <10% RSD. Standards should be within limits as established by control charts.

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### 3: Alkalinity by titration

Species Analysed: Alkalinity as bicarbonate  
Analyses provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences  
Method provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences

The alkalinity is measured by titrating the sample to a pH of 4.3 with a calibrated standard acid (H<sub>2</sub>SO<sub>4</sub>). A Metrohm auto-titration unit performed the analysis. The total alkalinity is reported as parts per million (ppm) HCO<sub>3</sub>.

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#### 4: Cations by Atomic Absorption

Species Analysed: Calcium, Iron, Manganese, Magnesium, Potassium, Sodium  
Analyses provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences  
Method provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences

The cations are analysed using atomic absorption spectrometry. The instrument utilized is a Varian Model 1475 Atomic Absorption Spectrophotometer. The instrument conditions are set using manufacturer's specifications.

The samples are filtered (0.45 µm) and acidified to a pH of 2 with nitric acid. The samples are stored at 4°C until they are analysed.

The in-house standards are commercially available and are compared to standards prepared by the Water Quality Laboratory. The standard are checked every five samples to monitor drift. The samples are run in duplicate. A set point standard is also analysed twice during a run of fifty samples. This assures the day to day quality of our in-house standards. The run is repeated if the in-house standards are not within five percent of the actual value.

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#### 5: Volatile Chlorinated Hydrocarbons by Gas Chromatography

Species Analysed: Carbon tetrachloride, chloroform, dichloroethene (all isomers), methylene chloride, tetrachloroethene, trichloroethene, vinyl chloride  
Analyses provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences  
Method provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences

## 5.1 Sample Preparation

Samples are collected in 22 mL glass vials, capped with tegrabond Teflon septa and aluminum seal (20 mm) and stored in a 4°C refrigerator until analysed. Prior to analysis they are decapped, 8 mL of sample quickly removed (with a 10 mL glass syringe) and the autosampler carousel.

Calibration standards are prepared by filling the same 22 mL vials with organic-free water and removing 8 mL from the total volume. Vials are quickly spiked with methanolic stocks and sealed.

## 5.2 Chromatographic Analysis: Automatic Headspace Analyser

The prepared samples and calibration spikes are run on a Hewlett Packard 5890 gas chromatograph

Table 2: GC conditions for the automated headspace procedure.

Part	Conditions
Column	DB-VRX 30 m X 0.32 mm I.D., 1.8 µm
Carrier	helium at 3.5 mL/min
Oven	isothermal at 32°C
Injector	Split 12:1, 150°C
Detector	PID (11.7 eV), 65°C; helium make up gas at 30 mL/min

equipped with a split injection port, capillary column, PID and a Varian Genesis headspace autosampler. Peak areas are measured by a HP 3392A integrator and an ESTD method of calibration is used.

Detection limits for these compounds are found to be 2-15 ppb (µg/L) using the EPA procedure for Method Detection Limit (MDL). The GC conditions are summarized in Table 2 above.

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## 6 Methanol Analysis by Direct Aqueous Injection

Species Analysed: Methanol

Analyses provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences

Method provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences

### **.6.1 Sample Preparation**

A 1.0 mL aqueous sample is placed in a 1.5 mL screw cap septum vial and sealed with a Teflon lined septa and screw cap. A 4  $\mu$ L aliquot of the aqueous solution is sampled for chromatographic analysis using a 10  $\mu$ L syringe equipped with a chaney adapter to enhance repeatability.

### **6.2 Chromatographic Analysis**

The aqueous samples are run on a Hewlett Packard 5840A gas chromatograph with a FID detector. The column is 10 ft. X 0.125 in. i.d., packed with 3% SP1500 on Carbopack B (80/100 mesh). The analyses are run isothermally at 100°C. A helium carrier gas at a flow rate of 20 mL/min is used. The detector temperature is 300°C and the injection temperature is 200°C.

Quantitative results are determined using an ESTD method of calibration and method detection limits for some of the compounds are found to be 100  $\mu$ g/L, using the EPA procedure for MDL.

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## 7: Dissolved Oxygen

### **7.1 Winkler Titration**

Species Analysed: Dissolved oxygen

Analyses provided by: Marianne Vandergrient, technician

Method provided by: The principles, limitations and reagent preparation for the Winkler method were discussed in detail by Lemon (1990) and Greenberg *et al.* (1985a).

### 7.1.1 Laboratory Analyses

- 1) Column input samples were obtained by diverting flow through the influent three way valve (Figure 3-3) into a 60 mL glass syringe.
- 2) Contents of the syringe were dispensed into a 60 mL hypo vial.
- 3) 0.6 mL of  $\text{MnSO}_4$  solution (480 g of  $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$  in 1 L solution) were added
- 4) 0.6 mL of alkali-iodide-azide reagent (500 g NaOH, 135 g NaI in 1 L solution, plus 10 g  $\text{NaN}_3$  in 40 mL solution).
- 5) Hypovial was shaken to mix the reagents and the sample, and the floc allowed to settle.
- 6) 0.6 mL of concentrated  $\text{H}_3\text{PO}_4$  solution was added and solution permitted to clear.
- 7) Sample was transferred to an Erlenmyer flask and a few drops of a starch indicator was added (4.0 g starch added to 200 mL of hot deionized water, stirred 5-10 minutes decanted into a plastic bottle, cooled overnight and preserved with a drop of toluene).
- 8) Sample titrated with 0.0025 N sodium thiosulphate standardized against potassium dichromate. The concentration of dissolved oxygen was then calculated from:

$$\text{mg/L} = \frac{(\text{volume } \text{Na}_2\text{S}_2\text{O}_3) (\text{concentration } \text{Na}_2\text{S}_2\text{O}_3 \text{ as mg/L})}{(\text{sample volume})} \quad (23)$$

### 7.1.2 Field Analyses

- 1) 60 mL polypropylene syringes were attached to the multilevel bundle sampling lines with a short length of Tygon tubing and approximately 10 mLs of groundwater were drawn into the syringe.
- 2) The Tygon tubing was clamped to prevent the groundwater from falling back down the sampling line, and the syringe removed. Any air entrapped in the syringe was then expelled and then the 10 mLs previously drawn were also expelled. The syringe was then reconnected to the sampling line. After this procedure, a continuous column of water was present from the syringe to the water table.
- 3) Slightly over 60 mL of water were drawn into the syringe and any entrapped gases (from degassing of the groundwater) were expelled.

- 4) The Winkler reagents ( $\text{MnSO}_4$ ,  $\text{NaI}$  and  $\text{H}_3\text{PO}_4$ ) were then added to the groundwater sample as described above.
- 5) The samples were stored on ice until delivered to the laboratory for the titration step. The total storage time did not exceed 24 hours.

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### 8: Dissolved Hydrocarbons (methane)

Species Analysed: Methane, ethane and ethene

Analyses provided by: Water Quality Laboratory, University of Waterloo, Department of Earth Sciences

Analyses provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences

Method provided by: Organics Laboratory, University of Waterloo, Department of Earth Sciences

#### 8.1 Sample Preparation

Samples are collected and stored in 60-100 mL screw cap bottles. A 15 mL aliquot is withdrawn from the bottom of the sample bottle into a 30 mL glass syringe and 15 mLs of air is added. The samples are shaken and allowed to equilibrate for a few hours. A 2.5 mL aliquot of the gas phase is injected for chromatographic analysis.

#### 8.2 Chromatographic Analysis

Samples are run on a Hewlett Packard 5840A gas chromatograph equipped with an FID and a 2.0

**Table 3:** GC conditions for the automated headspace procedure.

Part	Conditions
Column	30 m megabore GS-Q
Carrier	helium at 12 mL/min with a split vent at 50 mL/min
Oven	isothermal at 100°C
Injector	100°C
Detector	FID at 200°C

mL sample loop. The column is a 30 m megabore GS-Q. The analysis is run isothermally at 100°C.

The gas chromatograph is calibrated using an analysed gas mixture and values are reported as volume %. Dissolved concentrations are determined using the Ideal Gas Law, Henry's Law and solubility coefficients. The detection limit is estimated to be 5 µg/L.

## DISSOLVED HYDROCARBON ANALYSIS

(METHANE, ETHANE, ETHENE, PROPANE, PROPENE)

### Sample Preparation

Samples are collected in a screw cap glass container fitted with teflon-lined septa and stored at 4 degrees C for less than one week. A 15 ml aliquot is withdrawn from the sample bottle into a 30 ml glass syringe followed by 15mls of helium. The syringe is shaken and allowed to equilibrate for 3 hours. A 5-6ml aliquot of the gas phase is injected for chromatographic analysis.

### Chromatographic Analysis

The gas samples are analyzed with a Hewlett Packard 5840A gas chromatograph equipped with a flame ionization detector and a 2ml sample loop. The column is a 30 m megabore GS-Q. The analysis is run isothermally at 100 degrees C, with a helium carrier gas at a flow rate of 12 ml/min. The detector temperature is 200 degrees C and the injector temperature is 100 degrees C.

The gas chromatograph is calibrated in an external standard mode using several concentrations of analyzed gas mixtures (purchased from Praxair). The concentrations of the dissolved hydrocarbons in the original water samples are determined using the Ideal Gas Law, Henry's Law and solubility coefficients. The instrument detection limits (MDL) range from 0.02 to 0.09 ppb depending on the specific dissolved hydrocarbon.

Table 1- Quality Assurance Data

Compound	MDL ( $\mu\text{g/L}$ )
Methane	0.02
Ethene	0.09
Ethane	0.08
Propene	0.09
Propane	0.03

adsorption. Use recovery of model compounds representative of the various classes of organic halide compounds to evaluate validity of methods and instruments used.

4) Instrument calibration standard—Direct injection of trichlorophenol working standard onto the nitrate-washed method blank in concentrations over the working range of the instrument determines linearity and calibration of the analyzer module. After checking for proper microcoulometer function by injecting NaCl standard, pyrolyze duplicate instrument calibration standards and then duplicate method blanks. The net response to the calibration standards should be within 3% of the calibration curve value. Analyze additional instrument calibration standards after each eight sample pyrolyses and after cleaning the titration cell.

5) Calibration curve—Develop a standard curve by analyzing instrument calibration standards over the dynamic range of the microcoulometer. This dynamic range typically is from 0.5 to 50 µg chloride, but will vary between microcoulometers and titration cells. Because of the limited throughput of the TOX procedure, use single-instrument calibration standards at 50, 40, 30, 20, 15, 10, 5, 2.5, and 0.5 µg organic chloride to construct a standard curve after changes in an instrument's configuration, such as replacement of a titration cell or major instrument maintenance. Daily, analyze additional replicates of several instrument calibration standards to insure reproducibility, linearity, and proper function of the instrumentation and procedures. Select standard concentrations in the range of samples to be analyzed that day.

d. *Standard addition recovery:* During routine analyses, ideally make standard additions to every tenth sample. Recovery of 90% or more of the added amount indicates that the analyses are in control.

e. *Blanks:* High precision and accuracy of the background or blank value is important for accurate measurement of

TOX. Make blank measurements daily. Blanks that may be required are:

1) System blank—Analyze organic-free reagent water. The blank should have less than the minimum detectable concentration. Use this blank to insure that the equipment and procedures are not contributing to the TOX.

2) Method blank—Analyze GAC that has been nitrate-washed. Analyze duplicate method blanks daily before sample analysis and after each eight sample pyrolyses.

3) Standard blank—Analyze reagent water to determine the blank for standards.

4) Purgeable organic halogen blank—Analyze organic-free, pre-purged, reagent water to determine the POX blank.

f. *Sample duplicates:* To evaluate random bias analyze replicates of each sample. Establish acceptable control limits.

## 6. Calculation

Calculate the net organic chloride content ( $C_4$ ) of each filtered replicate of each sample and standard:

$$C_4 = \frac{C_1 - C_2 + C_3 - C_4}{V}$$

where:

$C_1$  = organic Cl on the first column or filter, µg.

$C_2$  = organic Cl on the second column or filter, µg.

$C_3$  = mean of method blanks on the same day and same instrument, µg Cl.

$C_4$  = uncorrected net organic Cl of filtered sample, µg organic Cl / L, and

$V$  = volume of sample filtered, L.

If applicable, calculate net purgeable organic Cl content ( $P_1$ ):

$$P_1 = \frac{P_1 - P_2}{V}$$

where:

$P_1$  = sample purgeable organic Cl, µg.

$P_2$  = blank purgeable organic Cl, µg.

$P_1$  = uncorrected net purgeable organic Cl, µg Cl / L, and  
 $V$  = volume of sample or standard purged, L.

Determine the linear regression of instrument calibration standard curves for each instrument configuration. Update this linear regression daily by including the standard points analyzed on that day. Calculate the corrected organic chloride concentration for each replicate of each sample by substituting the net organic chloride content ( $C_4$  or  $P_1$ ) of each sample replicate into the appropriate linear regression equation.

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## 507 OXYGEN DEMAND (BIOCHEMICAL)\*

### 1. Discussion

The biochemical oxygen demand (BOD) determination is an empirical test in which standardized laboratory procedures are used to determine the relative oxygen requirements of wastewaters, effluents, and polluted waters. The test measures the oxygen required for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulfides and ferrous iron. It also may measure the oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand) unless their oxidation is prevented by an inhibitor.

The method consists of placing a sample

in a full, airtight bottle and incubating the bottle under specified conditions for a specific time. Dissolved oxygen (DO) is measured initially and after incubation. The BOD is computed from the difference between initial and final DO.

The bottle size, incubation temperature, and incubation period are all specified. Most wastewaters contain more oxygen-demanding materials than the amount of DO available in air-saturated water. Therefore, it is necessary to dilute the sample before incubation to bring the oxygen demand and supply into appropriate balance. Because bacterial growth requires nutrients such as nitrogen, phosphorus, and trace metals, these are added to the dilution water, which is buffered to ensure that the pH of the incubation sample remains in a

\*Approved by Standard Methods Committee, 1981.

range suitable for bacterial growth. Complete stabilization of a sample may require a period of incubation too long for practical purposes; therefore, 5 d has been accepted as the standard incubation period.

Measurements of BOD that include both carbonaceous oxygen demand and nitrogenous oxygen demand generally are not useful; therefore, where appropriate, an inhibiting chemical may be used to prevent ammonia oxidation.<sup>1</sup> With this technique carbonaceous and nitrogenous demands can be measured separately. The inclusion of ammonia in the dilution water demonstrates that there is no intent to include the oxygen demand of reduced nitrogen forms in the BOD test. If this ammonia were oxidized, errors would result because the oxygen use would not be due exclusively to pollutants in the sample.

The extent of oxidation of nitrogenous compounds during the 5-d incubation period depends on the presence of microorganisms capable of carrying out this oxidation. Such organisms usually are not present in raw sewage or primary effluent in sufficient numbers to oxidize significant quantities of reduced nitrogen forms in the 5-d BOD test. Currently, many biological treatment plant effluents contain significant numbers of nitrifying organisms. Because oxidation of nitrogenous compounds can occur in such samples, inhibition of nitrification is recommended for samples of secondary effluent, for samples seeded with secondary effluent, and for samples of polluted waters.

The method included here contains both a dilution water check (5b) and a dilution water blank (5h). The dilution water check is to determine the acceptability of a particular batch of dilution water before it is used for BOD analysis. Seeded dilution waters are checked further for acceptable quality by measuring their consumption of oxygen from a known organic mixture, usually glucose and glutamic acid (5c).

The dilution water blank, made at the

same time that samples are analyzed, provides a further quality control on dilution water at the time of analysis as well as on the cleanliness of apparatus such as BOD bottles.

The procedure for determining immediate oxygen demand (IDOD) has been eliminated because: (a) it was not clear whether IDOD should be reported in 5-d BOD data; (b) the measurement was inaccurate because of the small differences between initial DO and DO after 15 min; (c) arbitrary selection of 15 min for measuring IDOD did not necessarily include all short-term oxygen-consuming reactions; and (d) the IDOD is, in some instances, an iodine demand (during the DO determination) rather than a true DO demand. The methods outlined here require determining initial DO immediately after making the dilution. In this way all oxygen uptake (including that occurring during the first 15 min) is included in the BOD measurement.

Although only the 5-d BOD is described here, many variations of oxygen demand measurements exist. These include using shorter and longer incubation periods, tests to determine rates of oxygen uptake, continuous oxygen uptake measurements by respirometric techniques, etc.

## 2. Sampling and Storage

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values. Minimize reduction of BOD by analyzing the sample promptly or by cooling it to near-freezing temperature during storage. However, even at low temperature, keep the holding time to a minimum. Warm the chilled samples to 20°C before analysis; some storage time can be used to accomplish this conveniently.

*a. Grab samples:* If analysis is begun within 2 h of collection, cooling is unnecessary. If analysis is not started within 2 h of sample collection, keep sample at or be-

low 4°C from the time of collection. Begin analysis within 6 h of collection; when this is not possible because the sampling site is distant from the laboratory, store at or below 4°C and report length and temperature of storage with the results. In no case start analysis more than 24 h after grab sample collection. When samples are to be used for regulatory purposes make every effort to deliver samples for analysis within 6 h of collection.

*b. Composite samples:* Keep samples at or below 4°C during compositing. Limit compositing period to 24 h. Use the same criteria as for storage of grab samples, starting the measurement of holding time from the end of the compositing period. State storage time and conditions as part of the results.

## 3. Apparatus

*a. Incubation bottles:* 250- to 300-mL capacity, with ground-glass stoppers. Clean bottles with a detergent, rinse thoroughly, and drain before use. As a precaution against drawing air into the dilution bottle during incubation, use a water-seal. Obtain satisfactory water seals by inverting bottles in a water bath or adding water to the flared mouth of special BOD bottles. Place a paper or plastic cup or foil cap over the flared mouth of the bottle to reduce evaporation of the water seal during incubation.

*b. Air incubator or water bath:* Thermostatically controlled at  $20 \pm 1^\circ\text{C}$ . Exclude all light to prevent possibility of photosynthetic production of DO.

## 4. Reagents

*a. Phosphate buffer solution:* Dissolve 8.5 g  $\text{KH}_2\text{PO}_4$ , 21.75 g  $\text{K}_2\text{HPO}_4$ , 33.4 g  $\text{Na}_2\text{HPO}_4 \cdot 7\text{H}_2\text{O}$ , and 1.7 g  $\text{NH}_4\text{Cl}$  in about 500 mL distilled water and dilute to 1 L. The pH should be 7.2 without further adjustment. Discard reagent (or any of the following reagents) if there is any sign of biological growth in the stock bottle.

*b. Magnesium sulfate solution:* Dissolve 22.5 g  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in distilled water and dilute to 1 L.

*c. Calcium chloride solution:* Dissolve 27.5 g  $\text{CaCl}_2$  in distilled water and dilute to 1 L.

*d. Ferric chloride solution:* Dissolve 0.25 g  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  in distilled water and dilute to 1 L.

*e. Acid and alkali solutions, 1N:* For neutralization of caustic or acidic waste samples.

*f. Sodium sulfite solution, 0.025N:* Dissolve 1.575 g  $\text{Na}_2\text{SO}_3$  in 1000 mL distilled water. This solution is not stable; prepare daily.

*g. Nitrification inhibitor:* 2-chloro-6-(trichloro methyl) pyridine.†

*h. Glucose-glutamic acid solution:* Dry reagent-grade glucose and reagent-grade glutamic acid at 103°C for 1 h. Add 150 mg glucose and 150 mg glutamic acid to distilled water and dilute to 1 L. Prepare fresh immediately before use.

## 5. Procedure

*a. Preparation of dilution water:* Place desired volume of water in a suitable bottle and add 1 mL each of phosphate buffer,  $\text{MgSO}_4$ ,  $\text{CaCl}_2$ , and  $\text{FeCl}_3$  solutions/L of water. Seed dilution water, if desired, as described in 5d. Test and store dilution water as described in 5b and 5c so that water of assured quality always is on hand.

*b. Dilution water check:* Use this procedure as a rough check on quality of dilution water. If dilution water has not been stored for quality improvement, add sufficient seeding material to produce a DO uptake of 0.05 to 0.1 mg/L in 5 d at 20°C. Do not seed dilution water that has been stored for quality improvement. Incubate a BOD bottle full of dilution water for 5 d at 20°C. Determine initial and final DO as in 5g and 5j. The DO uptake in 5 d at

†Nitrification Inhibitor 2533, Hach Chemical Co., or equivalent.

20°C should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.

If the oxygen depletion of a candidate water exceeds 0.2 mg/L obtain a satisfactory water by improving purification or from another source. Alternatively, if nitrification inhibition is used, store the seeded dilution water at 20°C until the oxygen uptake is sufficiently reduced to meet the dilution water check criteria. Storage is not recommended when BODs are to be determined without nitrification inhibition because nitrifying organisms may develop during storage. Check stored dilution water to determine whether sufficient ammonia remains after storage.

Before use bring dilution water temperature to 20°C. Saturate with DO by shaking in a partially filled bottle or by aerating with filtered air. Alternatively, store in cotton-plugged bottles long enough for water to become saturated with DO. Protect water quality by using clean glassware, tubing, and bottles.

*c. Glucose-glutamic acid check:* Because the BOD test is a bioassay the results can be influenced greatly by the presence of toxicants or by use of a poor seeding material. Distilled waters frequently are contaminated with copper; some sewage seeds are relatively inactive. Low results always are obtained with such seeds and waters. Periodically check dilution water quality, seed effectiveness, and analytical technique by making BOD measurements on pure organic compounds. In general, for BOD determinations not requiring an adapted seed, use a mixture of 150 mg glucose/L and 150 mg glutamic acid/L as a "standard" check solution. Glucose has an exceptionally high and variable oxidation rate but when it is used with glutamic acid, the oxidation rate is stabilized and is similar to that obtained with many municipal wastes. Alternatively, if a particular wastewater contains an identifiable major constituent contributes to the BOD, use

this compound in place of the glucose-glutamic acid.

Determine the 5-d 20°C BOD of a 2% dilution of the glucose-glutamic acid standard check solution using the techniques outlined in 5*d-j*. If the 5-d 20°C BOD value of the check is outside the range of  $200 \pm 37$  mg/L, reject any BOD determinations made with the seed and dilution water and seek the cause of the problem.

*d. Seeding:* It is necessary to have present a population of microorganisms capable of oxidizing the biodegradable organic matter in the sample. Domestic wastewater, unchlorinated or otherwise-undisinfected effluents from biological waste treatment plants, and surface waters receiving wastewater discharges contain satisfactory microbial populations. Some samples do not contain a sufficient microbial population (for example some untreated industrial wastes, disinfected wastes, high-temperature wastes, or wastes with extreme pH values). For such wastes seed the dilution water by adding a population of microorganisms. The preferred seed is effluent from a biological treatment system processing the waste. Where this is not available, use supernatant from domestic wastewater after settling at 20°C for at least 1 h but no longer than 36 h.

Some samples may contain materials not degraded at normal rates by the microorganisms in settled domestic wastewater. Seed such samples with an adapted microbial population obtained from the undisinfected effluent of a biological process treating the waste. In the absence of such a facility, obtain seed from the receiving water below (preferably 3 to 8 km) the point of discharge. When such seed sources also are not available, develop an adapted seed in the laboratory by continuously aerating a sample of settled domestic wastewater and adding small daily increments of waste. Optionally use a soil suspension or activated sludge to obtain the initial microbial population. Determine the exist-

ence of a satisfactory population by testing the performance of the seed in BOD tests on the sample. BOD values that increase with time of adaptation to a steady high value indicate successful seed adaptation. In making tests, use enough seed to assure satisfactory numbers of microorganisms but not so much that the oxygen demand of the seed itself is a major part of the oxygen used during incubation.

Determine BOD of the seeding material as for any other sample. This is the seed control. From the value of the seed control and a knowledge of the seeding material dilution (in the dilution water) determine seed DO uptake. To determine a sample DO uptake subtract the seed DO uptake from the total DO uptake. The DO uptake of the seeded dilution water should be between 0.6 and 1.0 mg/L.

Techniques for adding seeding material to dilution water are described for two sample dilution methods (§ 5).

*e. Sample pretreatment:*

1) Samples containing caustic alkalinity or acidity—Neutralize samples to pH 6.5 to 7.5 with a solution of sulfuric acid ( $H_2SO_4$ ) or sodium hydroxide (NaOH) of such strength that the quantity of reagent does not dilute the sample by more than 0.5%. The pH of seeded dilution water should not be affected by the lowest sample dilution.

2) Samples containing residual chlorine compounds—If possible, avoid samples containing residual chlorine by sampling ahead of chlorination processes. If the sample has been chlorinated but no detectable chlorine residual is present, seed the dilution water. If residual chlorine is present, dechlorinate and seed the dilution water (§5). Do not test chlorinated/dechlorinated samples without seeding the dilution water. In some samples chlorine will dissipate within 1 to 2 h of standing in the light. This often occurs during sample transport and handling. For samples in which chlorine residual does not dissipate in a rea-

sonably short time, destroy chlorine residual by adding  $Na_2SO_3$  solution. Determine required volume of  $Na_2SO_3$  solution on a 100- to 1000-mL portion of neutralized sample by adding 10 mL of 1 + 1 acetic acid or 1 + 50  $H_2SO_4$ , 10 mL potassium iodide (KI) solution (10 g/100 mL), and titrating with 0.025*N*  $Na_2SO_3$  solution to the starch-iodine end point. Add to the neutralized sample the volume of  $Na_2SO_3$  solution determined by the above test, mix, and after 10 to 20 min check sample for residual chlorine.

3) Samples containing other toxic substances—Certain industrial wastes, for example, plating wastes, contain toxic metals. Such samples often require special study and treatment.

4) Samples supersaturated with DO—Samples containing more than 9 mg DO/L at 20°C may be encountered in cold waters or in water where photosynthesis occurs. To prevent loss of oxygen during incubation of such samples, reduce DO to saturation at 20°C by bringing sample to about 20°C in partially filled bottle while agitating by vigorous shaking or by aerating with compressed air.

5) Sample temperature adjustment—Bring samples to  $20 \pm 1^\circ C$  before making dilutions.

6) Nitrification inhibition—If nitrification inhibition is desired add 3.33 mg 2-chloro-6 (trichloro methyl) pyridine to each bottle before capping or add sufficient amounts to the dilution water to make a final concentration of 10 mg/L. Samples that may require nitrification inhibition include, but are not limited to, biologically treated effluents, samples seeded with biologically treated effluents, and river waters. Note the use of nitrogen inhibition in reporting results.

*f. Dilution technique:* Dilutions that result in a residual DO of at least 1 mg/L and a DO uptake of at least 2 mg/L after 5 d incubation produce the most reliable results. Make several dilutions of prepared

sample to obtain DO uptake in this range. Experience with a particular sample will permit use of a smaller number of dilutions. A more rapid analysis, such as COD, may be correlated approximately with BOD and serve as a guide in selecting dilutions. In the absence of prior knowledge, use the following dilutions: 0.0 to 1.0% for strong industrial wastes, 1 to 5% for raw and settled wastewater, 5 to 25% for biologically treated effluent, and 25 to 100% for polluted river waters.

Prepare dilutions either in graduated cylinders and then transfer to BOD bottles or prepare directly in BOD bottles. Either dilution method can be combined with any DO measurement technique. The number of bottles to be prepared for each dilution depends on the DO technique and the number of replicates desired.

When using graduated cylinders to prepare dilutions, and when seeding is necessary, either add seed directly to dilution water or to individual cylinders before dilution. Seeding of individual cylinders avoids a declining ratio of seed to sample as increasing dilutions are made. When dilutions are prepared directly in BOD bottles and when seeding is necessary, add seed directly to dilution water.

1) Dilutions prepared in graduated cylinders—If the azide modification of the titrimetric iodometric method (Section 421B) is used, carefully siphon dilution water, seeded if necessary, into a 1- to 2-L-capacity graduated cylinder. Fill cylinder half full without entraining air. Add desired quantity of carefully mixed sample and dilute to appropriate level with dilution water. Mix well with a plunger-type mixing rod, avoiding entraining air. Siphon mixed dilution into two BOD bottles. Determine initial DO on one of these bottles. Stopper the second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, siphon dilution mixture into one BOD bottle. Determine initial DO on this

bottle and replace any displaced contents with sample dilution to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20°C.

2) Dilutions prepared directly in BOD bottles—Using a wide-tip volumetric pipet, add the desired sample volume to individual BOD bottles of known capacity. Fill bottles with enough dilution water, seeded if necessary, so that insertion of stopper will displace all air, leaving no bubbles. For dilutions greater than 1:100 make a primary dilution in a graduated cylinder before making final dilution in the bottle. When using titrimetric iodometric methods for DO measurement, prepare two bottles at each dilution. Determine initial DO on one bottle. Stopper second bottle tightly, water-seal, and incubate for 5 d at 20°C. If the membrane electrode method is used for DO measurement, prepare only one BOD bottle for each dilution. Determine initial DO on this bottle and replace any displaced contents with dilution water to fill the bottle. Stopper tightly, water-seal, and incubate for 5 d at 20°C.

g. *Determination of initial DO:* If the sample contains materials that react rapidly with DO, determine initial DO immediately after filling BOD bottle with diluted sample. If rapid initial DO uptake is insignificant, the time period between preparing dilution and measuring initial DO is not critical.

Use the azide modification of the iodometric method (Section 421B) or the membrane electrode method (Section 421F) to determine initial DO on all sample dilutions, dilution water blanks, and where appropriate, seed controls.

For activated sludge samples use either the membrane electrode method or the  $\text{CuSO}_4$ -sulfamic acid modification of the iodometric method (Section 421E). For muds use either the membrane electrode method or the alum flocculation modification of the iodometric method (Section 421D).

h. *Dilution water blank:* Use a dilution

water blank as a rough check on the quality of unseeded dilution water and cleanliness of incubation bottles. Together with each batch of samples incubate a bottle of unseeded dilution water. Determine initial and final DO as in 5g and 5f. The DO uptake should not be more than 0.2 mg/L and preferably not more than 0.1 mg/L.

i. *Incubation:* Incubate at  $20^\circ\text{C} \pm 1^\circ\text{C}$  BOD bottles containing desired dilutions, seed controls, dilution water blanks, and glucose-glutamic acid checks. Water-seal bottles as described in 5f.

j. *Determination of final DO:* After 5 d incubation determine DO in sample dilutions, blanks, and checks as in 5g.

## 6. Calculation

When dilution water is not seeded:

$$\text{BOD, mg/L} = \frac{D_1 - D_2}{P}$$

When dilution water is seeded:

$$\text{BOD, mg/L} = \frac{(D_1 - D_2) - (B_1 - B_2)f}{P}$$

where:

$D_1$  = DO of diluted sample immediately after preparation, mg/L,

$D_2$  = DO of diluted sample after 5 d incubation at 20°C, mg/L,

$P$  = decimal volumetric fraction of sample used,

$B_1$  = DO of seed control before incubation, mg/L,

$B_2$  = DO of seed control after incubation, mg/L, and

$f$  = ratio of seed in sample to seed in control = (% seed in  $D_1$ )/(% seed in  $B_1$ ).

If more than one sample dilution meets the criteria of a residual DO of at least 1 mg/L and a DO depletion of at least 2 mg/L and there is no evidence of toxicity at higher sample concentrations or the exist-

ence of an obvious anomaly, average results in the acceptable range.

In these calculations, corrections are not made for DO uptake by the dilution water blank during incubation. This correction is unnecessary if dilution water meets the blank criteria stipulated above. If the dilution water does not meet these criteria, proper corrections are difficult and results become questionable.

## 7. Precision and Accuracy

In a series of interlaboratory studies,<sup>2</sup> each involving 86 to 102 laboratories (and as many river water and wastewater seeds), 5-d BOD measurements were made on synthetic water samples containing a 1:1 mixture of glucose and glutamic acid in the total concentration range of 5 to 340 mg/L. The regression equations for mean value,  $\bar{X}$ , and standard deviation,  $S$ , from these studies were:

$$\bar{X} = 0.665 (\text{added level, mg/L}) - 0.149$$

$$S = 0.120 (\text{added level, mg/L}) + 1.04$$

For the 300-mg/L mixed primary standard, the average 5-d BOD was 199.4 mg/L with a standard deviation of 37.0 mg/L.

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## 508 OXYGEN DEMAND (CHEMICAL)\*

The chemical oxygen demand (COD) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to BOD, organic carbon, or organic matter. The test is useful for monitoring and control after correlation has been established. The dichromate reflux method is preferred over procedures using other oxidants because of superior oxidizing ability, applicability to a wide variety of samples, and ease of manipulation. Oxidation of most organic compounds is 95 to 100% of the theoretical value. Pyridine and related compounds resist oxidation and volatile organic compounds are oxidized only to the extent that they remain in contact with the oxidant. Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentration of free chloride ions.

### 1. Selection of Method

The open reflux method (A) is suitable for a wide range of wastes where a large sample size is preferred. The closed reflux methods (B and C) are more economical

in the use of metallic salt reagents, but require homogenization of samples containing suspended solids to obtain reproducible results. Ampules and culture tubes with premeasured reagents are available commercially. Follow instructions furnished by the manufacturer.

Determine COD values of > 50 mg O<sub>2</sub>/L by using procedures 508A.4a, 508B.4, or 508C.4. Use procedure 508A.4b to determine, with lesser accuracy, COD values from 5 to 50 mg O<sub>2</sub>/L.

### 2. Interferences and Limitations

Volatile straight-chain aliphatic compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when silver sulfate (Ag<sub>2</sub>SO<sub>4</sub>) is added as a catalyst. However, Ag<sub>2</sub>SO<sub>4</sub> reacts with chloride, bromide, and iodide to produce precipitates that are oxidized only partially. The difficulties caused by the presence of the halides can be overcome largely, though not completely, by complexing with mercuric sulfate (HgSO<sub>4</sub>) before the refluxing procedure. (Although 1 g HgSO<sub>4</sub> is specified for 50 mL sample, a lesser amount may be used where sample chloride con-

centration is known to be less than 2000 mg/L, as long as a 10:1 ratio of HgSO<sub>4</sub>:Cl<sup>-</sup> is maintained. Do not use the test for samples containing more than 2000 mg Cl<sup>-</sup>/L. Techniques designed to measure COD in saline waters are available.<sup>1,2</sup>

Nitrite (NO<sub>2</sub><sup>-</sup>) exerts a COD of 1.1 mg O<sub>2</sub>/mg NO<sub>2</sub><sup>-</sup>-N. Because concentrations of NO<sub>2</sub><sup>-</sup> in waters rarely exceed 1 or 2 mg NO<sub>2</sub><sup>-</sup>-N/L, the interference is considered insignificant and usually is ignored. To eliminate a significant interference due to NO<sub>2</sub><sup>-</sup>, add 10 mg sulfamic acid for each mg NO<sub>2</sub><sup>-</sup>-N present in the sample volume used; add the same amount of sulfamic acid to the reflux vessel containing the distilled water blank.

Reduced inorganic species such as ferrous iron, sulfide, manganous manganese,

etc., are oxidized quantitatively under the test conditions. For samples containing significant levels of these species, stoichiometric oxidation can be assumed from known initial concentration of the interfering species and corrections can be made to the COD value obtained.

### 3. Sampling and Storage

Preferably collect samples in glass bottles. Test unstable samples without delay. If delay before analysis is unavoidable, preserve sample by acidification to pH ≤ 2 using conc H<sub>2</sub>SO<sub>4</sub>. Blend samples containing settleable solids with a homogenizer to permit representative sampling. Make preliminary dilutions for wastes containing a high COD to reduce the error inherent in measuring small sample volumes.

## 508 A. Open Reflux Method

### 1. General Discussion

*a. Principle:* Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>). After digestion, the remaining unreduced K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> is titrated with ferrous ammonium sulfate to determine the amount of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> consumed and the oxidizable organic matter is calculated in terms of oxygen equivalent. Keep ratios of reagent weights, volumes, and strengths constant when sample volumes other than 50 mL are used. The standard 2-h reflux time may be reduced if it has been shown that a shorter period yields the same results.

### 2. Apparatus

*Reflux apparatus*, consisting of 500- or 250-mL erlenmeyer flasks with ground-

glass 24/40 neck\* and 300-mm jacket Liebig, West, or equivalent condenser† with 24/40 ground-glass joint, and a hot plate having sufficient power to produce at least 1.4 W/cm<sup>2</sup> of heating surface, or equivalent.

### 3. Reagents

*a. Standard potassium dichromate solution, 0.0417M:* Dissolve 12.259 g K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, primary standard grade, previously dried at 103°C for 2 h, in distilled water and dilute to 1000 mL.

*b. Sulfuric acid reagent:* Add Ag<sub>2</sub>SO<sub>4</sub>, reagent or technical grade, crystals or powder, to conc H<sub>2</sub>SO<sub>4</sub> at the rate of 5.5 g Ag<sub>2</sub>SO<sub>4</sub>/kg H<sub>2</sub>SO<sub>4</sub>. Let stand 1 to 2 d to dissolve Ag<sub>2</sub>SO<sub>4</sub>.

*c. Ferriin indicator solution:* Dissolve 1.485 g 1,10-phenanthroline monohydrate

\*Approved Standard Methods Committee, 1985.

\*Corning 5000 or equivalent.  
†Corning 2360, 9154 or equivalent.

## **APPENDIX I**

### **CALIBRATION CURVE ANALYSIS**

## Calibration Curve Analysis

The equations presented below were obtained from two sources: Anderson (1987) and Snedecor and Cochran (1989). Page references, for the various equations and definitions, from these two books are cited below using the first letters of the authors' names as abbreviations.

### Some definitions:

n	=	number of points in the regression line
m	=	number of points averaged to produce each point in the regression line
r	=	the correlation coefficient
t	=	the t statistic here representing $t_{0.05/2, n-2}$ in the general case and $t_{0.05/2, n-1}$ for the case where the curve is forced through the origin. In both cases this is appropriate for determining 95% confidence intervals.
X	=	the independent random variable
Y	=	the dependant variable
b	=	the slope
a	=	the intercept
$S_x^2$	=	variance of X values
$S_y^2$	=	variance of Y values
$S_{yx}$	=	the standard deviation of Y on X

### Formulas for some statistical parameters:

$$S_x^2 = \frac{\Sigma X^2 - \frac{(\Sigma X)^2}{n}}{n-1}$$

$$S_Y^2 = \frac{\Sigma Y^2 - \frac{(\Sigma Y)^2}{n}}{n-1}$$

A pg. 97 (2)

$$S_{yx} = \sqrt{\frac{(n-1)S_Y^2 - b^2 S_X^2}{n-2}}$$

A pg. 97 (3)

$$S_{(x-x)^2} = \Sigma X^2 - \frac{(\Sigma X)^2}{n} = \Sigma x^2$$

S&C pg. 151 (4)

$$C^2 = \frac{1}{S_{(x-x)^2}} \left( \frac{tS_{yx}}{b} \right)^2$$

S&C pg. 171 (5)

$$\bar{X} = \frac{\Sigma X}{n}$$

average X (6)

$$\bar{Y} = \frac{\Sigma Y}{n}$$

average Y (7)

Analogous definitions can be written for the case where the regression line is forced through the origin:

$$OS_{yx} = \sqrt{\frac{\Sigma Y^2 - \frac{(\Sigma XY)^2}{\Sigma X^2}}{n-1}}$$

(8)

A pg. 104, S&C pg. 174

$$OS_b = \frac{OS_{yx}}{\sqrt{\Sigma X^2}}$$

S&C pg. 174 (9)

With the above definitions, the following may be calculated:

1) the correlation coefficient:

$$r = \frac{n\sum XY - \sum X \sum Y}{\sqrt{(n\sum X^2 - (\sum X)^2)(n\sum Y^2 - (\sum Y)^2)}} \quad \text{A pg. 119 (10)}$$

2) the slope of the regression line (general case):

$$b = \frac{n\sum XY - \sum X \sum Y}{n\sum X^2 - (\sum X)^2} \quad (11)$$

A pg. 97, S&C pg. 151

3) the 95% confidence interval for the slope:

$$b = b \pm t_{0.05/2, n-2} \frac{S_{yx}}{\sqrt{\sum X^2 - \frac{(\sum X)^2}{n}}} \quad \text{A pg. 99 (12)}$$

$$a = \frac{\sum Y - b\sum X}{n} \quad \text{A pg. 97 (13)}$$

4) the intercept is given by:

5) the 95% confidence interval for the intercept:

$$a = a \pm t_{0.05/2, n-2} S_{yx} \sqrt{\frac{1}{n} + \frac{(\sum X)^2}{\sum X^2 - \frac{(\sum X)^2}{n}}} \quad \text{A pg. 99 (14)}$$

6) the t statistic ( $t_{cal}$ ) which is used to decide whether the line may be forced through the origin or not is given by:

$$t_{cal} = \frac{\bar{Y} - b\bar{X}}{S_{yx} \sqrt{\frac{1}{n} + \frac{\bar{X}^2}{S_{(x-x)^2}}} \quad (15)$$

A pg. 103, S&C pg. 175

If  $t_{cal} < t_{0.05/2, n-2}$  then the curve may be forced through the origin.

7) When the curve is forced through the origin, the intercept is 0, and the slope may be calculated from:

$$Oslope = b_{origin} = \frac{\sum XY}{\sum X^2} \quad (16)$$

A pg. 104, S&C pg. 174

8) The 95% confidence interval for the slope of a line forced through the origin is given by:

$$b_{origin} = b_{origin} \pm t_{0.05/2, n-1} OS_b \quad S\&C \text{ pg. } 174 \quad (17)$$

9) For the purposes of predicting Y, or graphing the calibration curves, the 95% confidence intervals for the dependant variable, Y, can be determined for

a) the general case:

$$Y = Y \pm t_{0.05/2, n-2} S_{yx} \sqrt{\frac{1}{m} + \frac{1}{n} + \frac{(X - \frac{\sum X}{n})^2}{S_{(x-x)^2}}} \quad (18)$$

A pg. 101, S&C pg. 166 (symbols in two references differ)

b) the case of the line forced through the origin:

$$Y = Y \pm t_{0.05/2, n-1} OS_{yx} \sqrt{1 + \frac{X^2}{\sum X^2}} \quad (19)$$

A pg. 101, S&C pg. 166

10) The 95% confidence intervals for the prediction of the independent variable, X, can be determined from:

$$X = \frac{\frac{Y - \bar{Y}}{b}}{1 - C^2} \pm \frac{t_{0.05/2, n-2} \frac{S_{yx}}{b} \sqrt{\frac{n+m}{nm} (1 - C^2) + \frac{(\frac{Y - \bar{Y}}{b})^2}{S_{(x-x)^2}}}}{1 - C^2} \quad (20)$$

S&C pg. 171 and top 172

11) The practical detection limit for the calibration curve may be determined from (21) by setting Y=0 and calculating the upper value for X at the 95% confidence level.

12) The +/- % may be calculated from the confidence interval as follows:

$$Err\% = \frac{|X_{upper} - X| + |X - X_{lower}|}{2X} * 100\% \quad (21)$$

13) The 95% confidence intervals for the prediction of the independent variable, X, when the line is forced through the origin can be determined from:

$$X = \frac{Y}{b} \pm t_{0.05/2, n-1} \frac{OS_{yx}}{b} \sqrt{1 + \frac{(\frac{Y}{b})^2}{\Sigma X^2}} \quad \text{A pg. 107 (22)}$$

**APPENDIX J**

**QAPP  
FOR THE EXTERNAL LAB**

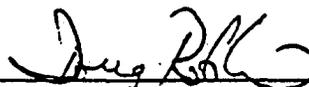
**INCHCAPE TESTING SERVICES  
ENVIRONMENTAL LABORATORIES  
SAN JOSE FACILITY**

**QUALITY ASSURANCE PROGRAM PLAN**

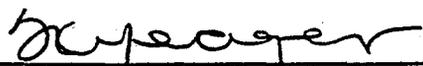
Revision 14.0

June, 1996

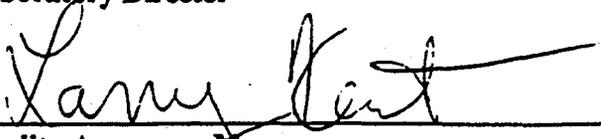
**Approval Signatures:**

  
\_\_\_\_\_  
**General Manager**

6/7/96  
\_\_\_\_\_  
**Date**

  
\_\_\_\_\_  
**Laboratory Director**

6-7-96  
\_\_\_\_\_  
**Date**

  
\_\_\_\_\_  
**Quality Assurance Manager**

6-7-96  
\_\_\_\_\_  
**Date**

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## **1.0 Quality Policy**

The objective of the quality assurance program at Inchcape Testing Services / Environmental Laboratories - San Jose is the generation of legally-defensible data through the use of documented, validated and standardized procedures.

This Quality Assurance Program Plan (QAPP) describes procedures to achieve the following:

- Maintain data integrity, validity and usability.
- Ensure analytical measurement systems are maintained in an acceptable state of stability.
- Detect problems through data assessment and establish corrective action procedures to assure a reliable analytical process.
- Document all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The management at Inchcape Testing Services is firmly committed to the Quality Assurance Program described in this plan. The laboratory's commitment to quality begins with the General Manager, who delineates policy and sets goals in conjunction with senior management personnel. Policies are implemented by management staff and laboratory personnel. All laboratory personnel are required to read and comply with this program.

A full-time Quality Assurance (QA) Department assists in the process of providing assessment of operating procedures along with recommendations for improvement.

The QA Department is headed by the QA Manager, who reports directly to the General Manager. The QA Manager oversees prevention, assessment and control procedures for the laboratory.

## **2.0 Organization and Management**

Inchcape Testing Services/ Environmental Laboratories is comprised of five laboratories in the United States and one in the United Kingdom. Each facility is under the supervision of an on-site General Manager. Laboratory General Managers report to the Chief Executive Officer of Inchcape Testing Services/ Environmental Laboratories.

Corporate quality policy is determined by the Quality Assurance Committee. The Committee is composed of the Quality Assurance Managers of each laboratory and is chaired by the corporate Technical Director. The Committee meets on a regular basis.

It is the policy of the laboratory that at each management and operational level a designated deputy or deputies will maintain continuity of service and other functions in the event of absence of key staff. The General Manager will ensure that all staff are made aware of their respective designated deputies and that they are fully aware of the extent and limitations of their responsibility.

It is the policy of the laboratory to discourage and reject all influence or inducements offered either by customers or suppliers which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the General Manager to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered staff are instructed to inform management immediately. It is the responsibility of the General Manager to take appropriate action to prevent recurrence.

Attached are the organization charts for the laboratory and the corporate staff of Inchcape Testing Services/ Environmental Laboratories. The quality assurance function at the laboratory is independent of laboratory operations. The Quality Assurance Manager reports directly to the General Manager of the facility. The Laboratory Director is responsible for the daily operations of the laboratory and also reports to the General Manager.

The General Manager bears the primary responsibility for data quality at the laboratory. The General Manager directs the functional areas of marketing, finance and administration for the laboratory.

The Laboratory Director is responsible for coordinating the activities of chemists, technicians and administrative support personnel. The Laboratory Director assures the commitment of sufficient resources for the timely generation of data of a known quality. The technical operation of the laboratory is the responsibility of the Laboratory Director.

The Quality Assurance Manager is responsible for the preparation and maintenance of the laboratory quality assurance program plan. The QA Manager acts as the official laboratory contact for audits, performance evaluation studies and project-specific quality control issues. The QA Manager approves and confirms the implementation of corrective actions for noncompliances. The QA Manager is responsible for the approval and distribution of controlled documents such as SOPs and the QAPP.

Department Supervisors and Managers are responsible for the overall flow of work and data through the laboratory. They are responsible for the maintenance of accurate SOPs and the enforcement of the QAPP and SOPs in their section to ensure that the data produced by the analysts is of a known quality and legally defensible. Further responsibilities include general management of all activities within their department; ensuring that all instrumentation and equipment meet performance criteria and calibration requirements; and training of laboratory staff. It is the responsibility of the Department Supervisor or Manager to ensure two levels of

data review prior to the release of data from the department. Department Supervisors inform the Laboratory Director of project status and capacity issues.

Project Managers act as liaisons between the laboratory and the client. Responsibilities include sample scheduling, communicating project-specific requirements to laboratory personnel, notifying the client of any sample receipt or analytical problems, monitoring the progress of analytical work, and providing validated results to clients in a timely manner. Project Managers respond to, document and resolve client complaints.

At the bench level, analysts are responsible for the generation of data by analyzing samples according to written SOPs. They are also responsible for ensuring that all documentation related to the analysis is complete and accurate. The analyst should inform the Department Supervisor or Manager of quality problems immediately. The analysts have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. Analysts are responsible for the initial review of all data.

Data Review personnel provide the final review of data following its release from the departments and the generation of the hardcopy reports. Calculations, calibrations and QC criteria are evaluated against the data quality objectives of the project or the laboratory QAPP. Any discrepancies found in the data should be reported to the appropriate Department Supervisor or Manager for corrective action.

Resumes of key personnel may be found in Appendix A.

### **3.0 Personnel**

It is the policy of the laboratory to engage permanent staff who are appropriately qualified and/or trained to perform their respective duties. Where, for commercial reasons, it is necessary to employ temporary staff, the laboratory shall ensure that the same criteria as those governing permanent staff apply with respect to training and qualifications.

Personnel training procedures begin with an established orientation program designed to familiarize the new associate with safety and chemical hygiene issues, the importance of QA/QC in the analytical laboratory, and company policies.

The level of training necessary to perform analytical tasks is derived from academic background and past experience, technical courses, and on-the-job training with specific methods or instrumentation. The responsibilities for formal academic training lie foremost with the individual. The responsibility for the additional specialized skills obtained through in-house training or external workshops is a shared obligation of the individual, their supervisor and the company. An associate's academic and professional experience are kept on file, including an initial statement of qualifications or resume and any additional documentation concerning subsequent training. Copies of certificates of completion, transcripts, diplomas or other documentation will be included in the files as appropriate.

New associates of all departments undergo the same orientation procedure. Laboratory personnel also undergo an extended basic training procedure involving videotapes covering basic laboratory skills. New associates must complete the viewing of these tapes within their first 90 days of employment. The basic videotape training covers the following subjects:

Safety	Titration	Use of Syringes
Pipetting	Representative Sampling	Laboratory Glassware
Weighing	Filtration	Math and the Metric System
pH Measurement	Understanding Data / SQC	General Lab Equipment

Inchcape Testing Services has a fundamental responsibility to provide facilities, equipment, maintenance and an organized program to make necessary improvements to ensure a safe working environment. The laboratory Health and Safety Manual is distributed to all new associates. This document provides a complete discussion of the safety policies enforced by the laboratory. This document will be reviewed annually and updated as necessary.

The San Jose facility is equipped with many structural safety features. Each associate must be familiar with the location, use and capabilities of general and specialized safety features associated with their work area. To protect associates from potential hazards, Inchcape Testing Services provides and requires the use of certain items of protective equipment. These include safety goggles, protective clothing, gloves, respirators, etc. For a complete description, please see the Health and Safety Manual.

The process of QA/QC training is an integral part of the analytical training. Trainees are under the supervision of experienced analysts, who are responsible for showing them the analytical procedures including applicable QA/QC measures. A new analyst will not be permitted to perform an analysis on client samples until their supervisor is confident that the analytical and QC procedures can be carried out correctly. Training documentation is included in the training file of each associate.

All associates are required to conform to the corporate ethics policy. A form, Misrepresentation of Data, is signed by each associate at the time of employment and annually thereafter. A copy of the form follows:

## Misrepresentation of Data

I understand that as part of my duties at Inchcape Testing Services/Environmental Laboratories, I have an obligation to produce timely and accurate analytical data. This requires that I maintain high standards of integrity so as to never compromise the quality of our work.

Accordingly, I understand that Inchcape Testing Services/Environmental Laboratories will strictly enforce a policy which prohibits, under any circumstances the willful misrepresentation of data. Willful misrepresentation means purposefully falsifying data or reporting false data. Examples of misrepresentation include, but are not limited to :

- Reporting false dates of preparation or analysis to meet holding times.
- Time traveling or resetting the computer acquisition clock (time or date) to meet holding times
- Changes in peak integration to meet QC control limits, including peak shaving or area adding.
- Changing instrument quantitation in the reporting software such as manual input of incorrect peak area counts.
- Falsifying instrument logbooks or run logs.

I understand that if I have any questions as to which course of action to take, or how to properly interpret data, I must consult appropriate lab supervisors or managers. Furthermore, I understand that if I feel pressured by another Associate or a client to misrepresent data or if I witness any co-workers misrepresenting data, I must immediately inform my supervisor, the QA Manager, and the Laboratory Director.

I understand that willful misrepresentation of data can result in serious consequences, both for the Associate and for Inchcape Testing Services. Associates may be prosecuted by local or federal authorities, be convicted of fraud, and face fines and even prison sentences. Inchcape may lose its reputation, certifications, and enough clients to severely damage our ability to continue as a business. I understand that under no circumstances can willful misrepresentation of data be tolerated from any Associate, at any level, or for any reason. Any act of willful misrepresentation of data may result in disciplinary action, up to and including termination of employment.

By signing below, I acknowledge that I have read and understood this communication and that I know of no misrepresentation of data by myself or any of my Associates. Furthermore, I acknowledge that I may be required to re-certify this commitment, at the Company's discretion, and that I will come forward immediately should any misrepresentation or attempt to misrepresent data come to my attention.

This document will be signed by all Associates of Inchcape Testing Services/Environmental upon commencement of employment and each year following.

\_\_\_\_\_  
signed

\_\_\_\_\_  
date

\_\_\_\_\_  
witnessed by

\_\_\_\_\_  
date

#### **4.0 Laboratory Facilities and Equipment**

Inchcape Testing Services - Environmental Laboratories is composed of facilities located throughout the United States and in the United Kingdom. The laboratories are located in San Jose, Baton Rouge, Boston, Burlington, Dallas and St. Helens, UK.

The San Jose facility was established in 1985 and offers a full-range of analytical services to the environmental community. The San Jose facility contains approximately 30,000 square feet of space for analytical and support services. Analytical laboratories comprise 13,600 square feet, while an additional 2,000 sq. ft. is devoted to sample receiving and storage. The remaining 15,000 sq. ft. is dedicated to administration, various support services and storage. A floor plan of the facility is included in this document. The laboratory is partitioned into functional group sections. Sufficient heating, ventilation and air conditioning systems provide adequate temperature control for personnel and equipment.

The laboratory is considered a secure facility. Access is controlled by locked doors, security codes and a staffed reception area. All visitors must sign in and be escorted by laboratory personnel while visiting the facility. The facility is protected by a burglar alarm, smoke detectors, gas leak sensors and a sprinkler system.

***Major Instrumentation and Equipment***

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**GAS CHROMATOGRAPHS/ MASS SPECTROMETERS**

- Hewlett-Packard 5972 Mass Spectrometer; Serial #: 3251A00161 (1992)  
➤ HP 5890 Series II Gas Chromatograph; Serial #: 3235A44699 (MSD3)  
➤ HP 7673 Autosampler; Serial #: 3240A32575  
➤ HP Chemstation Data Processing System
- Hewlett-Packard 5972 Mass Spectrometer; Serial #: 3050A02000 (1991)  
➤ HP 5890, Series II Gas Chromatograph; Serial #: 2750A17085 (MSD1)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 91143022  
➤ Dynatech PTA-30 Autosampler; Serial #: 10915-792E  
➤ HP Chemstation Data Processing System
- Hewlett-Packard 5971A Mass Spectrometer; Serial #: 3040A01385 (1991)  
➤ HP 5890A Gas Chromatograph; Serial #: 2950A28085 (MSD2)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 91168020  
➤ Dynatech PTA-30 Autosampler; Serial #: 10266-391B  
➤ HP Chemstation Data Processing System
- Hewlett-Packard 5972 Mass Spectrometer; Serial #: 3341A01394 (1994)  
➤ HP 5890 Series II Gas Chromatograph; Serial #: 2908A21554 (MSD6)  
➤ Archon 5100 Purge and Trap/Autosampler  
➤ HP Chemstation Data Processing System  
➤ Tekmar LSC 2000 Purge and Trap; Serial # 89125007
- Hewlett-Packard 5971 Mass Spectrometer; Serial #: 3304A04419 (1993)  
➤ HP 5890 Series II Gas Chromatograph; Serial #: 3310A47146 (MSD4)  
➤ HP 7673 Autosampler; Serial #: 2843A12442  
➤ HP Chemstation Data Processing System
- Hewlett-Packard 5972 Mass Spectrometer; Serial #: 3307A00625 (1994)  
➤ HP 5890 Series II Gas Chromatograph; Serial #: 3310A47588 (MSD5)  
➤ HP 7673 Autosampler; Serial #: 3009A2857  
➤ HP Chemstation Data Processing System
- Hewlett-Packard 5971A Mass Spectrometer (1995)  
➤ HP 5890 Series II Gas Chromatograph; Serial #: 27828A14504 (MSD7)  
➤ Archon 5100 Purge and Trap/Autosampler  
➤ HP Chemstation Data Processing System  
➤ Tekmar LSC 2000 Purge and Trap; Serial # 93194002

## GAS CHROMATOGRAPHS

*Volatile Analyses*

- Hewlett-Packard 5890A Series II Gas Chromatograph; Serial #: 0001340889 (1989)  
➤ OI 4560 Purge and Trap; Serial #: C301264 (HP15)  
➤ Dynatech PTA-30 Autosampler; Serial #: 10997-1092E  
➤ Electrolytic Conductivity Detector; Serial #: 89-958, 2720A70400  
➤ Photo-ionization Detector; Serial #: B325012, 2213-8-019
- Hewlett-Packard 5890A Series II Gas Chromatograph; Serial #: 0001310889 (1989)  
➤ OI 4460A Purge and Trap; Serial #: 522-7-057B (HP14)  
➤ Dynatech PTA-30 Autosampler; Serial #: 9828-890B  
➤ Electrolytic Conductivity Detector; Serial #: 90-1351, 5749-9-947  
➤ Photo-ionization Detector; Serial #: 3414530236, B414530239
- Hewlett-Packard 5890A Series II Gas Chromatograph; Serial #: 0001431090 (1990)  
➤ OI 4460A Purge and Trap; Serial #: 765-7-074A (HP24)  
➤ Dynatech PTA-30 Autosampler; Serial #: 7546-988  
➤ Electrolytic Conductivity Detector; Serial #: 1939-8-554, 6256-5-129
- Hewlett-Packard 5890A Series II Gas Chromatograph; Serial #: 0002510490 (1990)  
➤ HP 19395A Headspace Autosampler; Serial #: 2741I02426 (HP20)  
➤ Flame Ionization Detector
- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 0002500289 (1989)  
➤ HP 19395A Headspace Autosampler; Serial #: 2613I01232 (HP11)  
➤ Flame Ionization Detector
- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 002481286 (1989)  
➤ HP 19395A Headspace Autosampler; Serial #: 2741I01854 (HP3)  
➤ Flame Ionization Detector

*Pesticide Analysis*

- Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 3223A43534 (1992)  
➤ HP7673 Autosampler; Serial #: 2941A 20823 (HP25)  
➤ Dual Flame Ionization Detectors
- Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 2950A27078 (1990)  
➤ HP7673 Autosampler; Serial #: 3316A 31964 (HP18)  
➤ Flame Photometric Detector  
➤ Nitrogen Phosphorus Detector

- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2919A22602  
➤ HP7673 Autosampler; Serial #: 2847A12824 (1990)  
➤ Dual Electron Capture Detectors; Serial #: M1610, M1422 (HP16)
- Hewlett-Packard 5890A Gas Chromatograph (1989)  
➤ HP7673 Autosampler; Serial #: 3415A35307 (HP10)  
➤ Dual Electron Capture Detectors; Serial #: F1666, F4340
- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2623A08208 (1987)  
➤ HP7673 Autosampler; Serial #: 2919A14309 (HP5)  
➤ Dual Electron Capture Detectors; Serial #: F2125, L2735
- Hewlett-Packard 5890 Gas Chromatograph; Serial #: 2728A14504 (1987)  
➤ HP7673 Autosampler; Serial #: 3018A22404 (HP22)  
➤ Dual Electron Capture Detectors; Serial #: L1367, F5613
- Hewlett-Packard 5890 Gas Chromatograph (1987)  
➤ HP7673 Autosampler; Serial #: 34268A35907 (HP26)  
➤ Dual Electron Capture Detectors; Serial #: F4846, F4511
- Hewlett-Packard 5890 Gas Chromatograph (1995)  
➤ HP6890 Dual Tower Injector/ Autosampler (HP31)  
➤ Dual Electron Capture Detectors

#### *Petroleum Hydrocarbon Analyses*

- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2750A17377 (1990)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 90192008 (HP21)  
➤ Tekmar ALS2016 Autosampler; Serial #: 90206020  
➤ Flame Ionization Detector  
➤ Photo-ionization Detector; Serial #: A20045
- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2518A05297 (1986)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 90192011 (HP4)  
➤ Tekmar ALS2016 Autosampler; Serial #: 90163034  
➤ Flame Ionization Detector  
➤ Photo-Ionization Detector; Serial #: 920097
- Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2728A13404 (1987)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 89219019 (HP8)  
➤ Tekmar ALS2016 Autosampler; Serial #: 39236001  
➤ Flame Ionization Detector  
➤ Photo-ionization Detector; Serial #: 620030

Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2541A06765 (1986)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 89184006 (HP6)  
➤ Tekmar ALS2016 Autosampler; Serial #: 89172004  
➤ Flame Ionization Detector  
➤ Photo-ionization Detector; Serial #: 220033

Hewlett-Packard 5890A Gas Chromatograph; Serial #: 2908A21867 (1987)  
➤ Tekmar LSC 2000 Purge and Trap; Serial #: 88286008 (HP12)  
➤ Tekmar ALS2016 Autosampler; Serial #: 89165015  
➤ Flame Ionization Detector  
➤ Photo-ionization Detector; Serial #: 920051

Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 2950A27699 (1990)  
➤ HP 7673 Autosampler; Serial #: (Tray) 2942A20799; (Tower) 3009A20883 (HP19)  
➤ Flame Ionization Detector

Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 3336A56787 (1995)  
➤ HP 7673 Autosampler; Serial #: (Tray) 3449A37355; (Tower) 3448A40971 (HP27)  
➤ Flame Ionization Detector

Hewlett-Packard 5890 Series II Gas Chromatograph; Serial # 3336A57279 (1995)  
➤ HP 7673 Autosampler; Serial #: (Tray) 3449A37359; (Tower) 3508A41850 (HP29)  
➤ Flame Ionization Detector

Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 2518A04675 (1991)  
➤ HP 7673 Autosampler; Serial #: (Tray) 3032A22194; (Tower) 3013A22798 (HP23)  
➤ Flame Ionization Detector

Hewlett-Packard 5890 Series II Gas Chromatograph; Serial #: 2750A15437 (1987)  
➤ HP 7673 Autosampler; Serial #: (Tray) 2718A09201; (Tower) 2843A11415 (HP9)  
➤ Flame Ionization Detector

### *Organic Prep*

HP 5890, Series II Gas Chromatograph (1994)  
➤ HP Single-Injector Tower System (HP30)  
➤ Flame Ionization Detector

Hewlett-Packard Series 1050 High Pressure Liquid Chromatograph (1990)  
➤ Fluorescence Detector ; Serial #: 2702G0072 (HP17)  
➤ Ultraviolet Detector; Serial #: 2807G00210  
➤ HP1050 Autosampler; Serial #: 2941A00342  
➤ Pump; Serial #: 2840A00373

## Waters High Pressure Liquid Chromatograph

- Model 996 Photo Diode Array Detector
- Model 600 Pump
- Model 717 Autosampler
- Millenium Data Processing System

(1996)  
(LC2)

## INORGANICS

## Thermo Jarrell Ash ICAP61 Inductively Coupled Argon Plasma Spectrometer

- Simultaneous 35 Channel System Serial # 000607-0295
- TJA Autosampler
- Thermospec Data Acquisition and Control Software

(1988)  
(ICP1)

## Thermo Jarrell Ash ICAP61 Trace Analyzer Inductively Coupled Argon Plasma Spectrometer Serial # 000538-1293

- Simultaneous 31 Channel System
- TJA Autosampler Serial # 000375-0692
- Thermospec Data Acquisition and Control Software

(1993)  
(ICP2)

## Thermo Jarrell Ash ICAP61 Trace Analyzer Inductively Coupled Argon Plasma Spectrometer

- Simultaneous 31 Channel System
- TJA Autosampler
- Thermospec Data Acquisition and Control Software

(1996)  
(ICP3)

## Thermo Jarrell Ash Smith Hieftje 4000 Atomic Absorption Spectrophotometer Serial # 000357-0392

- TJA 188 Furnace Atomizer
- TJA AVA880 Hydride Generator
- TJA AS150 Autosampler
- Thermospec Data Acquisition and Control Software

(1992)  
(AA3)

## Thermo Jarrell Ash Smith Hieftje 4000 Atomic Absorption Spectrophotometer Serial # 000391-0792

- TJA 188 Furnace Atomizer
- TJA AS150 Autosampler
- Thermospec Data Acquisition and Control Software

(1991)  
(AA2)

## Leeman Labs PS200 Automatic Mercury Analyzer

- PS Data Acquisition Control Software

(1992)  
(HGA1)

## Leeman Labs PS200 II Automatic Mercury Analyzer

- PS Data Acquisition Control Software

(1996)  
(HGA3)

Applied Research Laboratories 902 Atomic Absorption Spectrophotometer	
➤ GBC GF2000 Graphite Furnace	(1987)
➤ GBC HG900 Hydride Generator	(AA1)
➤ GBC PAL2000 Autosampler	
Dionex Series 4500I Ion Chromatograph Serial # 000197-0190	(1990)
➤ Conductivity Detector	(IC1)
➤ Pulsed Amperometric Detector	
➤ Dionex Autosampler	
➤ AI450 Data Acquisition and Control System	
Shimadzu 5000C Soil/ Water TOC Analyzer	(1996)
➤ Solid Sample Module SSM-5000A	(TOC2)
➤ ASI 5000A Autosampler	
O.I. Corporation Model 700 TOC Analyzer	(1996)
	(TOC1)
Milton Roy Spectronic 1001	
➤ UV/VIS Spectrophotometer Serial # 000528-0993	(1992)
HF Scientific DRT-15C Nephelometer	(1990)
Fisher Accumet 50-pH, Conductivity, Salinity Meter	(1994)
Milton Roy Spectronic 21D Spectrophotometer Serial # 000198-0190	(1988)
Beckman Model TJ-6 Centrifuge (2)	(1992)
IEC Centra-4B Centrifuge	(1990)
Sartorius Analytic AC210 S Analytical Balance	(1993)
Brinkmann Digital Buret	(1993)
Cavro Autodiluter	(1993)

#### ADDITIONAL LABORATORY EQUIPMENT

- Perkin Elmer Fourier Transform Infrared Spectrometer Model 1600
- Glas-Col 3D Shakers (2)

- Pensky-Marten Closed Cup Flash Tester
- Tekmar Sonic Disrupters with Dual Converters (3)
- ABC 1002-A Gel Permeation & Chromatography Systems (2)
- Millipore Zero Headspace Extractors (12)
- Millipore Rotary Agitator
- Millipore Waste Filtration System
- Labconco WaterPro Polishing Station
- Millipore Milli-Q Water System
- Associated Design Manufacturing Rotary Extractors (2)
- Lars Lande Manufacturing Rotary Extractor
- Zymark Benchmate Gel Permeation Column Cleanup System
- Continuous Liquid-Liquid Extractors (72)
- Soxhlet Extractors (25)
- Orion 720 pH/ISE Meters (3)
- CEM MDS-2000 Microwave Sample Digestion System

## **5.0 Preventative Maintenance**

In order to prevent system downtime, minimize corrective maintenance cost and to help insure data validity, Inchcape employs a system of preventative maintenance. Operator manuals are used to pinpoint steps in the preventative maintenance scheme for individual instruments. All routine maintenance is performed as recommended by the manufacturer. The manuals also assist in the identification of commonly needed replacement parts, so that an inventory of these parts may be maintained at the laboratory. Routine maintenance is performed by the analyst, while external technicians may be called in for major repairs.

A bound maintenance logbook is associated with each instrument. Notation of the date and maintenance activity is recorded every time special or routine service procedures are performed. This includes routine service checks by laboratory personnel as well as factory service calls.

## 6.0 Document Control

Security and control of documents is necessary to insure that confidential information is not distributed and to make sure that all current copies of a given document are from the latest revision. Controlled documents have a header placed in the upper right hand corner of each page. This header provides enough information to unambiguously identify each page as part of a single compiled document. In some cases, the information may be distributed elsewhere in the margins of the page in order to facilitate readability, but, at a minimum the following information will be supplied:

Document Name  
Revision Number  
Date of Issue  
Page \_\_\_ of \_\_\_

Documents considered to be controlled include the laboratory's quality assurance plan and standard operating procedures (SOPs). These documents should be maintained in such a manner as to ensure traceability of all revisions and copies in circulation.

Documents are controlled through means of a colored stamp marking process combined with a circulation record. The following stamp is used to mark the approval pages plus any other pages considered critical to the integrity of the document:

### CONTROLLED DOCUMENT

Copy No. \_\_\_\_\_

**DO NOT DUPLICATE**

If this stamp is not colored red,  
this is not a controlled copy.

The unstamped originals of the controlled documents are maintained by the QA Manager. Every time a copy is made of a controlled document, it is stamped and tracked using the Document Control Tracking List Form. Revisions of documents are distributed to the original recipients of controlled copies as noted in the tracking list. All recipients will be requested to destroy out-dated revisions.

All laboratory logbooks are controlled by assigning each a unique identifier and recording the name of the recipient and its use in a bound logbook maintained by the QA Department. All bound logbooks shall be paginated.

## 7.0 Procedures for Traceability of Measurements

Balances are serviced on an annual basis by an external technician. This service is documented on each balance with a signed and dated calibration stamp. Balance calibrations are verified on a daily basis using Class 1 weights. Analytical balances are checked at multiple weights and the

measured weight is recorded in a bound logbook. Acceptance criteria is documented in each logbook.

Volumetric glassware used at the laboratory shall be Class A. The only exception to this requirement is the use of 1000 mL graduated cylinders for the measurement of samples for organic extraction. Volumetric glassware should not be exposed to extreme temperatures.

The calibration of automatic pipettors shall be carried out gravimetrically every six months at a minimum. This procedure shall be documented in a bound logbook. The calibration shall be verified and documented on a daily basis prior to use.

Traceability of measurements is assured through the use of a system of documentation and analysis of testing materials. All standards used in the calibration of instrumentation are certified by the supplier as to their accuracy. These certificates of analysis are maintained by the QA Department. The preparation of all standards is recorded in department Standard Preparation Logbooks. Information to facilitate traceability is included in this documentation. All standard and reagent labels must contain the following information: solution ID; concentration; date of preparation; initials of preparer; and expiration date.

The use of testing material from sources other than those of the standards used for calibration contribute to assuring the accuracy of measurements at the laboratory. Every initial calibration is verified through the use of secondary source calibration check.

## **8.0 Laboratory Scope of Tests**

The laboratory performs analyses for organic and inorganic parameters on a number of matrices. Analyses follow acceptable protocols approved under applicable state and federal programs. The laboratory is approved to provide testing in a number of states and under a number of programs. Please see Appendix H for the current laboratory certifications.

## **9.0 Arrangements Ensuring Laboratory Review of New Work**

For the laboratory to perform additional work within its scope or to expand its scope of testing a thorough review must be undertaken. Laboratory management considers available resources and current and pending workload prior to accepting new work.

It is the responsibility of the Laboratory Director, with input from the Department Heads and General Manager, to assess the ability of the laboratory to accept new work.

## **10.0 Reference to Test Procedures Used**

Methods performed at the laboratory may be found in the following references:

U.S. Environmental Protection Agency (EPA). "Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods." 3rd ed. Final Update II, 1995.

EPA. "Methods for the Chemical Analysis of Water and Wastewater." EPA-600/4-79-020, 1983.

EPA. "Methods for the Determination of Organic Compounds in Drinking Water" EPA/600/4-88/039. 1991.

EPA. "Methods for the Determination of Metals in Environmental Samples." EPA/600/4-91/010. 1991.

EPA. "Methods for the Determination of Metals in Environmental Samples. Supplement 1." EPA/600/R-94/111. 1994.

EPA. "Methods for the Determination of Inorganic Substances in Environmental Samples." EPA/600/R-93/100. 1993.

EPA. "Contract Laboratory Program (CLP) Statement of Work (SOW) for Inorganic Analysis, Multi-Media, Multi-Concentration." ILM04.0. 1994.

EPA. "CLP SOW for Inorganic Analysis, Multi-Media, Multi-Concentration." ILM03.0. 1992.

EPA. "CLP SOW for Inorganic Analysis, Multi-Media, Multi-Concentration." ILM02.1. 1991.

EPA. CLP SOW for Organic Analysis, Multi-Media, Multi-Concentration." OLM03.1. 1994.

EPA. CLP SOW for Organic Analysis, Multi-Media, Multi-Concentration." OLM01.9. 1991.

American Public Health Association (APHA). Standard Methods for the Examination of Water and Wastewater. 18th ed. 1992.

40 CFR, Part 136, Appendix A. "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater." 1984.

### **11.0 Procedures for Handling Test Items**

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. A documented and approved sampling plan reflecting data quality objectives should be in place at the sampling site. The integrity of the sample should be maintained through the use of preservation techniques specified in the relevant protocols (See Appendix D, Field Sampling Guide). Samples should be submitted to the laboratory under standard chain-of-custody procedures. A copy of the laboratory chain-of-custody form may be found in Appendix F.

Samples are received at the laboratory by a designated Sample Custodian. The Sample Custodian removes the samples from the cooler and compares the sample labels with the

information provided on the chain-of-custody form. If applicable, sample preservation, including temperature, is checked at the time of sample receipt. (Volatile water sample preservation is checked at the time of analysis.) A Sample Receiving Checklist (See Appendix E) is filled out for each group of samples received at the laboratory. The checklist is initialed and dated by the Sample Custodian and the Project Manager. If any nonconformances or discrepancies are noted on the checklist, a Corrective Action Form is generated. The Project Manager shall take appropriate action and document the resolution to any problems on the Corrective Action Form.

General information for each group of samples is entered into a bound logbook and assigned a workorder number (e.g. 9601001). Each sample within the workorder is assigned a unique Inchcape Testing Services ID number (Workorder No. + sample number, e.g. 9601001-01). A label with this number is placed on each container for the sample.

After the samples are labeled they are placed into the appropriate sample storage area. Sample containers for volatile analysis are placed in refrigerators in the volatile laboratories, while sample containers for inorganic and semivolatile organics analysis are placed in the walk-in refrigerators. Temperatures of sample storage areas are maintained at 4 +/-2 C and are monitored on a daily basis by the QA Department. The temperatures are recorded in a bound logbook. Out of control incidents and corrective action are documented in the bound temperature logbook as well. Cross-contamination of samples in the volatile sample storage areas is checked by the analysis of storage blanks on a weekly basis. The QA Manager will be notified immediately if any target compounds are detected at levels above the reporting limit in the storage blanks.

The following information is then entered into the laboratory sample tracking system:

Client name, address, phone number	Report recipient's name
Workorder number	P.O. Number (if required)
Project Number or Name	Due Date (verbal and hardcopy)
Laboratory ID for each sample	Client Sample ID
Method(s)	Date sampled
Sample container location	Comments

After the information is entered into the system, it is reviewed by the Sample Control Supervisor. The workorder is then released and the information is available to laboratory personnel who have access to the system. A secondary review of the log-in information is carried out by the Project Manager to ensure compliance with project requirements.

Sample movement within the laboratory is recorded on internal chains-of-custody associated with each workorder. These documents are provided to the client in extended data packages or if requested.

Every effort is made to ensure a representative aliquot is removed from the sample container. Homogenization of the sample may be carried out when appropriate, particularly in trace

metals analysis. For volatile analysis of soils, the topmost layer of soil is removed prior to obtaining an aliquot for analysis. Water samples for volatile analysis must be without headspace. The laboratory requests a minimum of three vials for volatile analysis. This allows the laboratory to screen samples and, if any containers contain bubbles the laboratory can avoid using those for final analysis.

Samples are stored for a minimum of thirty days after the final report is issued. A longer storage time may be requested by the client. Sample disposal is then carried out following applicable state and federal guidelines.

## **12.0 Records**

The laboratory will retain all records related to sample analysis including raw test data, calculations, derived data, calibrations and test reports.

These records shall be stored in a systematic manner for a minimum of seven years. Longer periods of storage may be arranged at the time of project initiation.

Mistakes should never be erased, deleted or written over. They shall be corrected by drawing a single line through the error and entering the correction alongside. The correction shall then be initialed and dated by the responsible person.

Original observations should be entered into bound logbooks or onto properly designated work sheets. In most cases these work sheets shall be bound at the time of use, however some forms, such as sample preparation forms, may be bound after use when sufficient numbers have been generated.

## **13.0 Procedures for Calibration and Verification**

Calibration and verification procedures are detailed in method SOPs. These procedures are generally specific to instrumental technique and environmental program. Prior to the use of any method at the laboratory, an approved SOP must be in place.

Methods performed at the laboratory must be validated prior to sample analysis. Method validation involves the determination of sensitivity; and linearity and reproducibility studies. Method sensitivity is determined by method or instrument detection limit studies. The procedure to determine the method detection limit (MDL) follows 40 CFR, Part 136, Appendix B. The reporting limit for a given analyte may be derived from the MDL. In general, the quantitation or reporting limit for an analyte is 3-10 x MDL.

A determination of the linearity of the instrument response may be performed through the analysis of multiple standards at increasing concentrations or by analyzing a single high level standard. If multiple standards are analyzed an assessment of their linear response is required. This may be carried out by regression analysis or by the calculation of calibration factors.

When determining the upper level of linearity for a method, the absence of analyte carryover must be verified.

#### 14.0 Verification Practices

The laboratory participates in a number of proficiency studies. These studies may be single- or double-blind. Twice annually the laboratory analyzes samples from both the EPA's Water Pollution (WP) and Water Supply (WS) Performance Evaluation Studies. On a quarterly basis the laboratory participates in the corporate double-blind performance evaluation study. Furthermore, a solid matrix PE sample is analyzed twice annually in single-blind studies.

All unacceptable results shall be addressed in written corrective action responses submitted to the laboratory QA Department. The adequacy of the corrective action may be verified through the successful analysis of follow-up PE samples.

The retesting of retained samples shall be carried out on a periodic basis. Comparison of results should take into account possible discrepancies due to time between analyses. This is particularly important with volatile organic compounds.

The laboratory performs quality control samples on an ongoing basis. These requirements and acceptance criteria are outlined below in Section 15.0.

#### 15.0 Corrective Action

Analytical Procedure	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
All	Method Detection Limit (MDL) Study	Annually for water and soil.	Minimum of 7 replicates. Spike level must be at a concentration between the calculated MDL and 10 x MDL.	Reprepare study at adjusted spiking levels for MDLs falling outside of acceptable range.
	Initial Demonstration of Proficiency	Prior to use of method for client samples.	Method-specific. Precision and accuracy goals established for many parameters in published methods.	Investigate failure. Verify solution integrity and instrument performance. Reprepare and reanalyze.

<b>All (cont'd)</b>	Ongoing Demonstration of Proficiency	Annually for each analyst.	Method-specific goals to ascertain analysts ability to perform method.	Investigate failure and repeat performance test. Repeated failures may indicate need for additional training.
<b>Inorganics - GFAA, CVAA, Wet and IC</b>	Initial Calibration.	Daily.	Minimum of 3 points and a blank. Correlation coefficient $\geq 0.995$ .	Check instrument performance. Verify solution integrity.
	Initial Calibration Verification (ICV).	Following initial calibration.	Secondary source at a concentration other than that used in initial calibration. Must be within +/- 10% of expected value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Recalibrate instrument.
	Initial Calibration Blank (ICB)	Following ICV.	Target analytes at less than absolute value of reporting limit.	Check instrument performance and perform necessary maintenance. Check reagents for contamination. Recalibrate.
	Continuing Calibration Verification (CCV)	Following ICV, after every 10 analyses and at the end of the analytical sequence.	At a concentration other than ICV. Must be within +/- 10% of expected value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Recalibrate instrument and reanalyze all samples from last acceptable CCV.

<b>Inorganics - GFAA, CVAA, Wet, IC(cont'd)</b>	Continuing Calibration Blank (CCB)	Following each CCV, after every ten analyses and at the end of the analytical sequence.	Absolute value of all target analytes must be less than reporting limit.	Investigate source of contamination. Reanalyze all samples since last acceptable CCB.
	Reporting limit standard (CRA). Required for CLP. Not required when low standard of initial calibration is at reporting limit.	Following ICV, but prior to sample analysis, and at end of run, or every 8 hr.	Spike at reporting limit for all methods. Results within +/- 35% of true value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Reanalyze once, if still out recalibrate and reanalyze all samples since last acceptable CRA.
	Laboratory Control Sample (LCS).	One per preparation batch.	All spiked elements must be +/- 20% of true value.	Reanalyze once. Verify solution integrity and instrument performance. Reprepare and reanalyze all associated samples for failed analyte(s).
	Matrix Spike (MS)	One per 20 field samples of a similar matrix.	All spiked elements must meet method- specific control limits.	Reanalyze once. Verify solution integrity and instrument performance. Reprepare and reanalyze MS. If still out, perform Post- Digestion Spike (PDS).

<b>Inorganics - GFAA, CVAA, Wet, IC(cont'd)</b>	Sample Duplicate (D) or Matrix Spike Duplicate (MSD)	One per 20 field samples of a similar matrix.	All elements at concentrations greater than 5 x reporting limit must be +/- 20% RPD for water and +/- 35% RPD for soil.	Reanalyze sample and duplicate. Reprepare sample and duplicate, if still out, flag data and narrate.
<b>Inorganics (ICP)</b>	Initial Calibration.	Daily.	Minimum of 3 points and a blank. Correlation coefficient $\geq 0.995$ . CLP requires a minimum of one standard and a blank.	Check instrument performance. Verify solution integrity. Recalibrate instrument.
	Initial Calibration Verification (ICV).	Following initial calibration.	Secondary source at a concentration other than that used in initial calibration. Must be within +/- 10% of expected value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Recalibrate instrument.
	Initial Calibration Blank (ICB)	Following ICV.	Target analytes at less than absolute value of reporting limit.	Investigate source of contamination. Recalibrate instrument
	Continuing Calibration Verification (CCV)	Following ICV, after every 10 analyses and at end of analytical sequence.	At a concentration other than ICV. Must be within +/- 10% of expected value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Recalibrate instrument and reanalyze all samples from last acceptable CCV.

<b>Inorganics (ICP) (cont'd)</b>	<b>Continuing Calibration Blank (CCB)</b>	Following each CCV and after every ten analyses. Must be run to close out analytical run.	Absolute value of all target analytes must be less than reporting limit.	Investigate source of contamination. Reanalyze all samples from last acceptable CCB.
	Reporting limit standard (CRI). Required for CLP. Not required when low standard of initial calibration is near reporting limit.	Following ICV, but prior to sample analysis, and following the analysis of last sample, or every 8 hr, whichever is more frequent.	Spike at 2 x reporting limit for all analytes. Results within +/- 35% of true value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Reanalyze once, if still out recalibrate and reanalyze all samples since last acceptable CRI.
	Interference Check Sample (ICS)	Following ICV, but prior to sample analysis, and following the analysis of last sample, or every 8 hr, whichever is more frequent.	All analytes present must be within 20 % of expected value.	Check instrument performance and perform necessary maintenance. Verify solution integrity. Reanalyze all samples since last acceptable ICS.

<b>Inorganics (ICP) (cont'd)</b>	Laboratory Control Sample (LCS).	One per preparation batch.	All spiked elements must be +/- 20% of true value.	Reanalyze once. Verify solution integrity and instrument performance. Reprepare and reanalyze all associated samples for failed analyte(s).
	Matrix Spike (MS)	One per 20 field samples of a similar matrix.	All spiked elements must meet method- specific control limits.	Reanalyze once. Verify solution integrity and instrument performance. Reprepare and reanalyze MS. If still out, perform Post- Digestion Spike (PDS).
	Sample Duplicate (D) or Matrix Spike Duplicate (MSD)	One per 20 field samples of a similar matrix.	All elements at concentrations greater than 5 x reporting limit must be +/- 20% RPD for water and +/- 35% RPD for soil.	Reanalyze sample and duplicate. Reprepare sample and duplicate, if still out, flag data and narrate.
<b>Organics (GC/MS)</b>	Tuning	Every 12 hr, prior to analysis.	Method specific criteria. See specific SOP.	Inspect instrument and perform necessary maintenance. Retune instrument and reanalyze any samples since last acceptable tune.
	Breakdown and Tailing Check	Daily, prior to analysis of samples.	DDT breakdown ≤ 20% and no tailing evident for PCP and Benzidine.	Perform system maintenance. Repeat check.

<b>Organics (GC/MS) (cont'd)</b>	<b>Initial Calibration</b>	As necessary.	Minimum of five points. Low standard must not exceed method reporting limit. See specific SOP or SOW for acceptance criteria.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
	<b>Secondary Source Calibration Verification</b>	Following every initial calibration.	Secondary source check - all target compounds +/- 20% of expected value.	Verify solution integrity and instrument performance. Reanalyze, if still out, then recalibrate.
	<b>Continuing Calibration Verification</b>	Every 12 hr.	Please see applicable SOP or SOW for acceptance criteria.	Verify solution integrity and instrument performance. Reanalyze standard once, if still out recalibrate and reanalyze affected samples.
	<b>Method Blanks</b>	For extractable methods, one per preparation batch. For purgeable methods, one every 12 hr.	Target compounds < reporting limit, except for common lab contaminants (e.g. methylene chloride, phthalates) Common lab contaminants may exceed the reporting limit to a degree specified in the applicable SOP or SOW	Investigate source of contamination. Reprepare and reanalyze all associated samples.

<b>Organics (GC/MS) (cont'd)</b>	Surrogate Spike	Every sample, blank and standard.	Method-specific limits, please see SOP or applicable SOW.	Check calculations. If still out, verify instrument performance and correct, if necessary. Reanalyze sample, if still out reextract and reanalyze, report both sets of data.
	Laboratory Control Sample	One per analytical batch.	Method specific control limits. Please see SOP or SOW.	Reanalyze once, if still out verify solution integrity and instrument performance. Reprepare and reanalyze all associated samples.
	Matrix Spike (MS) and Matrix Spike Duplicate (MSD)	One set per 20 samples of a similar matrix.	Method-specific limits. Please see SOP or applicable SOW.	Reanalyze once, if still out verify solution integrity and instrument performance. If LCS acceptable, narrate as possible matrix effect.
	Instrument Blanks	Following high level samples and standards.	Target compounds < reporting limits.	Continue to analyze instrument blanks or perform decontamination procedures.

<b>Organics - GC and HPLC</b>	<b>PEM</b>	Daily, prior to analysis of samples.	DDT and Endrin breakdown $\leq 20\%$ .	Perform system maintenance. Repeat column check.
	<b>Initial Calibration</b>	As necessary.	Minimum of five points. Low standard must not exceed method reporting limit. %RSD may not exceed 20% for all target analytes.	Verify solution integrity and check instrument performance. Perform necessary maintenance and recalibrate instrument. Reanalyze all affected samples.
	<b>Secondary Source Calibration Verification</b>	Following every initial calibration.	Secondary source check - all target compounds +/- 20% of expected value.	Verify solution integrity and instrument performance. Reanalyze, if still out, then recalibrate.
	<b>Retention Time (RT) Window Study</b>	Every new column installation	All target compounds and surrogates in all standards must fall within calculated window. RT is calculate from 3 x s of three standards run over a period of 72 hr.	Perform system maintenance. Reanalyze affected samples.
	<b>Continuing Calibration Verification</b>	Every 10 samples and following sample analysis to close out bracket.	Please see applicable SOP or SOW for acceptance criteria.	Verify solution integrity and instrument performance. Reanalyze standard once, if still out recalibrate and reanalyze affected samples.

<b>Organics - GC and HPLC (cont'd)</b>	<b>Method Blanks</b>	For extractable methods, one per preparation batch. For purgeable methods, one every analytical run, up to a maximum of 20 samples.	Target compounds < reporting limit, except for common lab contaminants (e.g. methylene chloride, phthalates) Common lab contaminants may exceed the reporting limit to a degree specified in the applicable SOP or SOW	Investigate source of contamination. Reprepare and reanalyze all associated samples.
	<b>Surrogate Spike</b>	Every sample, blank and standard.	Method-specific limits, please see SOP or applicable SOW.	Check calculations. If still out, verify instrument performance and correct, if necessary. Reanalyze sample, if still out reextract and reanalyze, report both sets of data.
	<b>Laboratory Control Sample</b>	One per analytical batch.	Method specific control limits. Please see SOP or SOW.	Reanalyze once, if still out verify solution integrity and instrument performance. Reprepare and reanalyze all associated samples.
	<b>Matrix Spike (MS) and Matrix Spike Duplicate (MSD)</b>	One set per 20 samples of a similar matrix.	Method-specific limits. Please see SOP or applicable SOW.	Reanalyze once, if still out verify solution integrity and instrument performance. If LCS acceptable, narrate as possible matrix effect.

<b>Organics - GC and HPLC (cont'd)</b>	Instrument Blanks	Following high level samples and standards.	Target compounds < reporting limits.	Continue to analyze instrument blanks or perform decontamination procedures.
	Second column confirmation	All positive hits above reporting limit for GC and HPLC.	Within method holding time. Concentrations on two columns should agree within 25%.	Inspect for interferences. Report lower of two values and narrate. If difference between two columns > 25%, flag with "P".

### 16.0 Departures from Policies

Departures from laboratory Standard Operating Procedures are not permitted unless the approval of the Laboratory Director and/ or Quality Assurance Manager is obtained prior to implementation of the departure. These exceptions must be documented and signed off. Documentation shall be archived with the specific project information. If a client requests a departure from the laboratory's policies or procedures, the Project Manager shall notify the QA Manager, who will then discuss the steps necessary to implement such a departure with the Laboratory Director and appropriate Department Manager/ Supervisor. Written approval will be forwarded to the Project Manager.

If the proposed deviation from an approved SOP originates internally, an Analytical Method Change Form (See Appendix G) must be completed. The change will be reviewed by the QA Manager to evaluate compliance with published methodology and laboratory policies. The form must then be signed off by both the QA Manager and the Department Manager/ Supervisor. The change will be incorporated into the appropriate SOP and a new revision will be generated and distributed. This departure cannot be implemented until after the Analytical Method Change Form has been signed off.

### 17.0 Procurement and Inventory Control

Chemical reagents, solvents, gases, glassware and general chromatographic supplies are ordered as needed to maintain sufficient quantities on hand. Purchase orders are maintained as an inventory control of materials ordered by the laboratory. All orders are processed through the receiving department and distributed to the appropriate departments.

The grade or purity ordered varies depending on analytical requirements. All reagents and standards must meet ACS Reagent Grade or better. Chemical reagents and standards are dated when received and/or prepared in the laboratory and stored according to manufacturer's

recommendations. Flammable cabinets are utilized for the storage of flammable solvents and reagents. Chemicals in glass shall be double-contained to prevent uncontrolled spills. Incompatible chemicals are stored separately.

Solvents and reagents used in the preparation of samples are purchased on a lot basis following analysis, and approval by the QA Department. Records of the lot checks are maintained by the department supervisors.

The purchase of analytical instrumentation is based on anticipated sample volume and the need to maintain data quality. Specifications are carefully examined to be sure new instrumentation meets current and anticipated needs. Warranty and service contract information is gathered at the time bids are reviewed and is considered in the final purchasing decision. An extensive performance check-out before the instrument is accepted is mandatory. Operators of new instruments are sent to training courses if necessary.

### **18.0 Procedures for Addressing Complaints**

Circumstances which raise doubt about the quality of the laboratory's data or its compliance with stated policies may be considered complaints. Complaints may originate internally or externally and may or may not be in written form. It is important that complaints be documented and tracked. Complaints will be addressed during internal audits.

Generally, all external complaints are received and handled by the Project Manger (PM) assigned to the client. Once a complaint is received, it is recorded in the PM's bound telephone logbook. It is then left to the discretion of the PM whether or not it is necessary for a supervisor or laboratory management to become involved in responding to the client. The occurrence of the complaint must be brought to the attention of the QA Manager. The QA Manager will determine if the circumstances leading to the complaint warrant further investigation. If it is determined that further action is necessary, an audit of the activity or circumstance will be conducted.

Internal complaints should be directed to appropriate management personnel. Issues raised relating to data quality or the quality system in general shall be brought to the attention of the QA Manager. A file of these complaints will be maintained. These complaints will be addressed during internal audits.

### **19.0 Client Confidentiality**

It is laboratory policy not to release any information pertaining to projects, except to the client who submitted the samples or who was responsible for billing.

Prior to the release of any information to a third party, written consent must be obtained by the laboratory from the original client. This release may be transmitted via facsimile, but must be on the client company letterhead.

Sensitive documentation from internal and external sources, when appropriate, shall be shredded before being discarded

## **20.0 Audit and Review**

Two types of audits are performed at the laboratory. These are technical system audits and data audits. Technical system audits are designed to assess the adequacy of a selected system in meeting its objectives. Data audits are performed to assess the quality of results reported to clients. The laboratory will notify affected clients promptly, in writing, when the audit findings cast doubt on the correctness or validity of the laboratory's test results.

Technical system audits are designed to assess the adequacy of a selected system in meeting its objectives and will be performed on a quarterly basis, or as needed. Audit reports will be submitted to laboratory management. Corrective action responses should be provided in a timely manner and will be verified by follow-up audits.

Data audits are performed on a quarterly basis. Approximately 100 results, that have been released by the laboratory, are randomly selected by the QA Manager. The results are evaluated for methods, units and accuracy. The results of the audit are evaluated and submitted to the corporate Quality Assurance Committee.

A review of the quality system by management shall be carried out annually. The goal of this review is to ensure that the laboratory's quality system is adequate for the achievement of the laboratory's quality objectives and to introduce any necessary changes or improvements. This review must be documented and any actions resulting from it should be implemented within a reasonable amount of time. At a minimum the review must address the following: matters arising from the previous review; reports from A2LA and client audits; results of internal audits, including corrective actions implemented; results of participation in proficiency testing; results of in-house quality checks; resolution of internal and external complaints; staff training; adequacy of staff, equipment and facility resources; and future plans and estimates for new work, staff and equipment.

## **21.0 Subcontracting of Tests**

Samples subcontracted by the laboratory shall only be sent to laboratories that are competent to perform the tests requested at a level equivalent to the requirements of ISO Guide 25, General Requirements for the Competence of Calibration and Testing Laboratories. Where feasible the sub-contractor shall be assessed by the laboratory either through an on-site visit or by the submission of sufficient documentation to determine the sub-contractor's capabilities and qualifications. This documentation may include, but is not limited to, QAPP, SOPs, recent PE sample results and relevant certifications. Such documentation shall be filed at the laboratory.

An Intercorporate Work Form (Appendix I) shall be used when subcontracting tests within Inchcape Testing Services/ Environmental Laboratories.

## 22.0 Computer Hardware and Software

Wherever possible the laboratory will establish standards for computer systems and peripheral equipment. In instances where a vendor-provided solution is bundled with hardware and software, the vendor will certify that the proposed hardware will readily operate with existing hardware platforms, and will provide operating and maintenance instructions. Computer system hardware will be configured only by IT associates or trained vendor technicians. A log will be maintained for each major item of hardware. Major items of hardware include systems used for data collection, multi-user file servers, and multi-user printers.

Prior to release for production use of any in-house developed software, the software is considered to be under development. Software must be validated prior to release. Validation of software consists of testing the output of the software, based on sample input data, and comparing the output with independently calculated results. After software has been tested and is approved for release, copies of the source and any executable file images are placed under configuration control. Control of the source code and related executable files is maintained by the IT Director or designee. No changes are made to production software without making an assessment of the impact of the new revision and without formal approval. When changes have been approved, the original source code and executable images are archived in a library prior to the installation of the updated version.

## **APPENDIX A - RESUMES OF KEY PERSONNEL**

### **DOUGLAS M. ROBBINS - CHIEF EXECUTIVE OFFICER/VICE PRESIDENT**

#### **EDUCATION:**

M.B.A., Finance, University of San Francisco, San Francisco, California, 1986.  
B.S., Atmospheric and Oceanic Science, University of Michigan, College of Engineering; Ann Arbor, Michigan; 1973.

#### **PROFESSIONAL EXPERIENCE:**

**Chief Executive Officer** - Mr. Robbins directs the functional areas of marketing, finance, and administration for the Anametrix, Inc. Environmental Testing Laboratory. He has broad based experience in the management of environmental service firms and extensive knowledge of state and federal environmental regulations. His operational responsibilities include program management, contract administration, and client liaison.

**Assistant Vice President** - Corporate wide sales and marketing management for this international environmental consulting and laboratory services firm. Established large national accounts in the industrial, commercial and financial services sectors. Trained, developed and guided the regional sales staff in the attainment of sales goals. Directed all marketing activities, including technical conferences, seminars, advertising, and creation of promotional literature. Clayton Environmental Consultants, Inc., Pleasanton, California, 1989 through 1991.

**Vice President, Regional Manager** - Managed a staff of 35 employees through effective delegation of responsibility. Directed regional and corporate planning processes and business development strategies. Prepared annual budgets and capital expenditure plans. Provided controlled growth in excess of 25 percent per year. Med-Tox Associates, Inc., Pleasant Hill, California, 1986 through 1989.

**Vice President** - Corporate responsibilities in the areas of marketing, finance and administration. Established and implemented corporate marketing objectives and strategies. Overall responsibility for cash management and expense control. Developed administrative procedures for effective organizational control and inter-branch operations. Curtis & Tompkins, Ltd., San Francisco, California, 1982 to 1986.

**Branch Manager** - Start-up and management of the California based western regional laboratory for this national environmental corporation. Environmental Research Group, Inc., Emeryville, California, 1979 to 1982.

**Branch Manager** - Successful start-up of the company's first branch laboratory, demonstrating analytical proficiency to become the first commercial laboratory to obtain certification by the Ohio EPA. Environmental Research Group, Inc., Cleveland, Ohio, 1975 to 1979.

**Project Manager** - Performed a broad range of wet-chemical and instrumental analyses. Obtained experience with and theoretical knowledge of atomic absorption, gas chromatography, and neutron activation. Field Manager for a two year project with the International Joint Commission formed by Michigan, Ohio, Wisconsin, and Canada. Environmental Research Group, Inc., Ann Arbor, Michigan, 1972 to 1975.

## **SUSAN KRASKA YEAGER - LABORATORY DIRECTOR**

### **EDUCATION:**

B.A., Chemistry, Minor Biology, San Jose State University, 1985.

### **EXPERIENCE:**

**Laboratory Director** - Ms. Yeager is responsible for coordinating the activities of chemists, technicians and administrative support personnel, and assures that sufficient resources are available for the generation of consistently accurate and timely analytical data. She also continues to act as Program Manager for Government Contracts.

**Program Manager, Government Contracts** - Ms. Yeager has the responsibility to ensure project-specific requirements are met by laboratory personnel. She reviews data and diskettes for technical completeness before they are released to the client. She is the primary contact for all communication between the Anametrix laboratory and the contractor's project management, and assists in bid package proposals for large, multi-year contracts. Anametrix, Inc., San Jose, California, January 1992 to present.

**Manager, GC/MS, HPLC and Organic Preparation Departments** - Ms. Yeager had the responsibility for training chemists in the areas of GC/MS and HPLC operations and maintenance, CLP and SW-846 protocol requirements and data interpretation, sample scheduling and throughput, data review and introducing new methods to the departments such as air toxics (TO1, TO2, VOST, and carb 430). Mid-Pacific Environmental Laboratory (formerly Acurex), Mountain View, California, October 1989 to December 1991.

**R&D Supervisor** - Responsibilities included supervising chemists working on EPA method development projects, specifically the development of improving EPA's SW-846 methods, utilizing GC/MS, GC and HPLC. Worked with manufacturers to provide beta-test site for equipment and products evaluation for future applications. She was involved in marketing new areas and proposal preparation to promote department growth and visibility. Acurex Corp., Mountain View, California, March 1988 - October 1989.

**Senior Materials and Process Engineer** - Was responsible for setting up program and obtaining DHS Certification for the analysis of VOA and SVOA by GC/MS, and then training chemists to continue these procedures. Provided technical help and consultation to scientists and engineers from six internal corporate organizations for problems related to failures of materials or equipment. Used GC/MS and data from instruments like NMR, FTIR, ESCA,

and SEM to characterize contaminants and chemical products. Lockheed Missiles and Space Co., Sunnyvale, California, March 1986 - March 1988.

**Staff Chemist** - Set up new instrumentation including GC/MS and HPLC systems, responsible for the analysis of soil, water and waste using GC/MS, HPLC and GC with selective detectors. Developed and validated new methods for EPA projects, and was project chemist for several EPA method validations, SAS and large client projects. Acurex Corp., Mountain View, CA April 1980 - March 1986.

**R&E Technician** - Responsibilities included running samples and testing prototype equipment under development, such as the 4000-TSQ and the 4000-LC/MS belt interface. Provided assistance to R&E engineers with instrument specifications and maintenance. Finnigan Instruments, Sunnyvale, California, October 1979 - April 1980.

**Applications Technician** - Responsibilities included assisting in customer demonstrations of Finnigan instruments, sample preparation, packed column and capillary column preparation, ion source and other GC/MS maintenance of instruments used by the applications chemists. She also assisted in preparation for trade shows. Finnigan Instruments, Sunnyvale, California, August 1977 - October 1979.

## **MICHAEL HOBAN - INORGANICS MANAGER**

### **EDUCATION:**

Ph.D., Oceanography, The University of Texas, Austin, 1979.  
B.S., Chemistry and Molecular Biology, University of Illinois at Chicago, 1972.

### **EXPERIENCE:**

**Inorganics Manager** - Supervision of six chemists in the Inorganics Department performing wet chemistry, sample preparation, and instrumental analysis by AA, ICP and ion chromatography. Responsible for project scheduling, review of reports, instrument maintenance and troubleshooting, training, and method development. Inchcape Testing Services, San Jose, California, June 1995 to present.

**Inorganics Development Specialist** - New instrumentation and method development for Inorganics including sample preparation, analysis and wet chemistry. Development of diskette deliverables and automated reporting. Special projects, including the purchase and installation of the TJA 61E Trace Analyzer. Inchcape Testing Services, Anametrix Laboratories, San Jose, California, May 1993 to June 1995.

**Inorganics Manager** - Responsibilities include directing all functions of the Inorganics Laboratory including sample preservation, sample digestion and sample analysis. Analyses include total recoverable and dissolved metal determination for surface, ground water, extracts, waste, sediment, sludge and soil samples according to EPA approved methods. Instruments include 902 GBC Atomic Absorption, System 2000 graphite furnace with PAL 2000 and 900 hydride generation system, TJA 61 simultaneous inductively coupled plasma spectrophotometer with auto sampler TJA-4000 graphite furnace. Anametrix Inc., San Jose, California. January 1991 to April 1993.

**Director of Engineering Services** - Was responsible for the management of a certified environmental analysis laboratory and a technical writing department. Designed and supervised the renovation of the environmental laboratories, specified and purchased the analytical equipment (Ion Chromatograph, Inductively Coupled Argon Plasma Emission Spectrometer, Cold Vapor Atomic Absorption Spectrometer, Scanning Electron Microscope, Flame Atomic Absorption Spectrometer, Flow Injection Analyzer, Infrared Spectrophotometer, Gas Chromatograph, and classical wet chemistry equipment). Hired staff, established an independent QA/QC program, and designed a PC based laboratory information management system (LIMS). Directed the qualifying program for EPA and California DOHS certification.

Familiarity with the current State of California and EPA environmental regulations, including CERCLA, EPCRA (SARA-TITLE III), NPDES, RCRA, TSCA, and TITLE 22. Garratt - Callahan Company, San Francisco, California, 1985 to 1990.

**Tilton Research Fellow in Systematics** - California Academy of Sciences, Geology Department, 1984 to 1985.

**Research Specialist in Aquatic Toxicology** - Designed and managed multidisciplinary environmental analyses programs as a project manager and environmental consultant in aquatic and oceanic systems, including siting studies for industrial facilities, NPDES monitoring programs, aquatic toxicology bioassays, remediation programs for the mining industry, design and construction of aquatic toxicology test facilities, and environmental cost-benefit analyses for government and industry. Also organized the phytoplankton collection program for numerous oceanographic cruises, including the Islas Orcadas 17-78 Antarctic cruise and for the AMERIEZ, Antarctic Marine Ice-Edge Experiment. University of California at Berkeley, Botany Department, 1984.

**Visiting Lecturer, Marine Sciences** - University of California at Santa Cruz, Center for Coastal Marine Studies, 1983.

**Project Manager, Policy and Environmental Analysis Department**, Radian Corporation, 1979 through 1982.

**Research Associate** - Authored numerous papers, participated in international research and symposia, presented invited seminars, taught at the university level. Texas A&M University, Oceanography Department, 1976 to 1979.

## PROFESSIONAL ASSOCIATIONS

American Chemical Society  
American Association for the Advancement of Science  
National Association of Environmental Professionals  
American Society for Limnology and Oceanography

## **LAWRENCE M. KENT - QUALITY ASSURANCE MANAGER**

### **EDUCATION:**

B.S., Natural Resource Studies, University of Massachusetts, 1979.  
Graduate Study, Ecology, University of California, Davis, 1979-1983.

### **EXPERIENCE:**

**Quality Assurance Manager** - Review data and methodologies for quality assurance. Maintain Standard Operating Procedures. Conduct internal quality control audits. Oversee certification procedures and external quality control audits. Supervise Quality Assurance staff. Anametrix, Inc., San Jose, California, August 1989 to present.

**Quality Assurance Officer** - Review data and methodologies to ensure reliability of results and compliance with regulatory guidelines. Conduct internal quality control audits. Maintain control charts and other related documentation. Anametrix, Inc., San Jose, California, December 1987 to August 1989.

**Post-Graduate Researcher III** - Conducted research on the effects of air pollutants on various aspects of plant physiology and growth. This work included the construction and monitoring of outdoor fumigation structures, periodic sampling and analysis, statistical analyses, and report-writing. Department of Land, Air and Water Resources; University of California, Davis, January-December, 1984.

**Post-Graduate Researcher II** - Managed laboratory and research projects in the Vegetable Crops Seed Physiology Laboratory at the University of California, Davis, June 1982-July, 1983.

**Research Assistant** - Conducted experiments leading to Master's thesis. Work included atomic absorption spectrophotometry, radioisotope handling and analysis, and data analysis. Department of Land, Air and Water Resources, University of California, Davis, April 1980-May, 1982.

### **PROFESSIONAL ASSOCIATIONS:**

American Society for Quality Control

## **JIM HARPER - DATA REVIEW MANAGER**

### **EDUCATION:**

B.S., Chemistry, Botany; University of Florida, Gainesville, Florida  
M.S., Botany; University of Florida, Gainesville, Florida

### **EXPERIENCE:**

**Data Review Manager** - Responsible for overseeing the data review department. Assists with Quality Assurance matters pertaining to the quality of data. Inchcape Testing Services; San Jose August 1995 - present.

**QA Officer, Safety Officer** - Implemented federal programs level quality assurance program resulting in successful participation in the Navy's NFESC and Army Corps of Engineers programs. Developed formalized health and safety program emphasizing training and compliance to written safety plans, reducing the laboratory's workman compensation liability. Reorganized and managed hazardous waste management program, reducing the laboratory's regulatory liability. Mid-Pacific Laboratory; Mountain View, CA. 1991 to August 1995.

**QA Officer** - Implemented federal programs level quality assurance program, meeting the requirements of the Air Force HAZWRAP program and DOE's NQA-1 standard. IT Analytical Services; San Jose, CA. 1987 - 1991

**Safety Officer** - Managed health and safety program in compliance with corporate guidelines and federal and state OSHA requirements. IT Analytical Services; San Jose, CA. 1987 - 1990

**Group Leader** - Supervised project-dedicated analytical support team providing quick turnaround GC analyses for a large industrial remediation project. IT Analytical Services; San Jose, CA. 1986 - 1987.

**Project Manager, Research Chemist** - Managed project characterizing vapor emissions from power plant cooling towers. Managed project investigating occupational exposure to oil barge cargo emissions. Responsible for experimental design, test equipment fabrication, staff coordination analysis, data interpretation, fiscal control, and report writing. Participated in a number of research projects in the fields of analytical, environmental, and material sciences. SRI International; Menlo Park, CA. 1980 - 1986.

**Assistant Group Leader, Field Team Leader** - Led field sampling team for industrial and federal environmental assessment projects. Coordinated laboratory analyses for Navy base selection environmental impact study. Environmental Science and Engineering, Inc. 1976 - 1977.

## **CECIL CLASPELL - INFORMATION TECHNOLOGY DIRECTOR**

### **EDUCATION:**

B.S., Business, University of Phoenix, 1989.  
Utah State University, Major: Computer Science.  
Colorado State University, Major: Computer Science.

### **EXPERIENCE:**

**IT Director** - Responsibilities include overall operational business management of in-house information system and technology (software and hardware), management of LIMS systems, in-house accounting, report generation system, and business applications. Inchcape Testing Services; San Jose, CA. August 1994 to present.

**Senior Systems Analyst** - Responsible for requirements analysis, data collection, table loading, and installation of a multi-site Laboratory Information Management System (LIMS). UNIX System administrator for a 10-system IBM RS-5000 installation; INGRES Database administrator; Multi-LIMS administrator. Westinghouse Hanford Company; Richland, WA. July 1993 to August 1994.

**Programmer/Analyst** - As LIMS Project Manager, performed systems analysis related to selection of a LIMS for an Industrial Hygiene laboratory. Successfully install and configured the selected LIMS. Provided systems analysis, programming, and LIMS administration support during installation and operation of the LIMS. UNIX System administrator; IBM RS-6000; INGRES Database administrator; Multi-LIMS administrator. Hanford Environmental Health Foundation; Richland, WA. May 1991 to July 1993.

**Program Administrator** - Tested new hardware and software and provided technical assistance and product information to end users. Specified and approved hardware and software staff of 3000+ and instructed users on compliance with Federal regulations regarding ADPE acquisitions. Battelle Pacific Northwest Laboratories; Richland, WA. June 1990 to May 1991.

**Programmer/Analyst** - Developed a Laboratory Results Archive System, application software to operate an existing LIMS software, utilizing RMS databases, software on personal computers and VAX 11/750, and designed and implemented vendor reporting and time charge tracking systems. U.S. Testing Company; Richland, WA. August 1989 to June 1990.

**Engineering Scheduler/Programmer** - Developed computer software for Project Engineering Department, designed and wrote ThioCall

telecommunications program, and scheduled engineering activities using Project/2.

**ADDITIONAL ACTIVITIES:**

President, International Multi-LIMS User group.

Member, Who's Who of International Business Leaders

## TIM ALBRECHT - PESTICIDES MANAGER

### EDUCATION:

B.S. Chemistry, Florida Institute of Technology, Melbourne, FL; June 1984  
A.S. Chemistry, Lansing Community College, Lansing MI; June 1980

### EXPERIENCE:

**Pesticides Manager** - Responsibilities include instrument repair and maintenance, ordering supplies and spare parts, scheduling sample analyses, review of analytical results with clients, and method development. Inchcape Testing Services/ Environmental Laboratories. San Jose, CA; March 1996 to present.

**Operations Manager** - Direct supervision of the operations of the facility with shared accountability for management and profitability. Directed the technical aspects of the laboratory. Directly responsible for laboratory production and effective use of resources. Responsible for providing technical leadership and staff development as well as coordinating strategies concerning operation. Analytical Technologies, Inc. San Diego, California; September 1995 to January 1996.

**Quality Assurance Officer** - Coordinated, recommended, and assessed programs involving company quality assurance goals and quality control efforts. Reviewed results of existing quality assurance and quality control procedures. Recommended and implemented improvements to existing procedures. Managed both the internal and corporate performance evaluation programs. Team leader for facility audits at other corporate laboratories. Interacted with staff on matters concerning data quality and the transmission of data to company clientele. Encotec, Inc. Ann Arbor, MI; August 1994 to September 1995.

**Group Leader** - Supervised the GC/HPLC Laboratory comprising eleven chemists, twelve gas chromatographs, and one HPLC. Responsible for all aspects of lab operation and personnel management including instrumentation maintenance, final data review, timely delivery of data, budget analysis and cost control, personnel issues, and the overall direction of the lab. Project manager for one of the plant facilities. Encotec, Inc. Ann Arbor, MI; January 1990 to August 1994.

**Assistant Group Leader** - Assisted with the supervision of the GC/HPLC Lab while performing and reviewing the analyses of other chemists. Responsibilities mirrored those of Group Leader but focused on review of analytical data, instrument maintenance, and training. Encotec, Inc. Ann Arbor, MI; January 1989 to January 1990.

**Senior Chemist** - Supervised the EPA CLP pesticide group while learning all other analyses used in the GC/HPLC Lab. Reviewed and approved data. Responsible for teaching new chemists instrumental analysis including theory, maintenance, and optimization of the instruments. Encotec, Inc. Ann Arbor, MI; June 1987 to January 1989.

## **CATHERINE RAE MILTENBERGER - ORGANIC PREP MANAGER**

### **EDUCATION:**

B.A., Chemistry, California State University, Stanislaus, 1987.

### **EXPERIENCE:**

**Organic Prep Manager** - Oversees Organic Prep Department. Responsible for scheduling sample extractions and analyses. Trains prep chemists and technicians in GLP and EPA methodologies. Updates SOPs, maintains laboratory equipment and reviews all data submitted by the Organic Prep Laboratory. Anametrix, Inc., San Jose, California, November 1992 to present.

**Organic Prep Manager** - Responsible for determining capacity, scheduling work, data review, training and method development. Mid Pacific Environmental Lab, Mountain View, California, August 1991 to October, 1992.

**Organic Prep Lead Chemist** - Extracted soil and water samples for organics by EPA methods. Used GPC, alumina and acid cleanups. Responsible for preparing standards for spiking and analysis, training new employees, method development and writing SOPs. Acurex / Mid Pacific Environmental Lab, Mountain View, California, February 1989 to April, 1990.

**GC/Extraction Chemist** - Assisted in the GC Lab, loading samples and making dilutions. Extracted water and soil samples using EPA methods. California Water Laboratory, Modesto, California, 1987 - 1988.

## **SIA J. HOSSEINIAN - GAS CHROMATOGRAPHY/VOA SUPERVISOR**

### **EDUCATION:**

B.S., Chemistry, San Jose State University, San Jose, California, May 1992.

### **EXPERIENCE:**

**Gas Chromatography/VOA Supervisor** - Responsible for project scheduling, review of reports and instrument maintenance. Supervision of two chemists and two technicians in the analysis of volatile organics, using EPA Methods 601/602 and 8010/8020. Anametrix Inc., San Jose, California, October 1993 to Present.

**Pesticides Laboratory Chemist II**- Analysis of water and soil samples using EPA methods 608/8080, 614/8140, 612/8120, 632, 610/8310, 504/8011, and according to EPA CLP procedures. Responsible for scheduling work and meeting QA/QC requirements. Anametrix Inc., San Jose, California, February 1993 to October 1993.

**GC Chemist** - Analysis of soil and water samples according to EPA Methods 601/602 and 8010/8020 including instrument calibration, data processing and report generation. Anametrix, Inc., San Jose, California, March 1990 to February 1993.

**GC Technician** - Analysis of soil and water samples for gasoline with BTEX by purge and trap with gas chromatography PID/FID in series. Analysis for extractable hydrocarbons as diesel by GC/FID. Anametrix Inc., San Jose, California, August 1987 to February 1990.

**Sample Custodian** - Receipt and log-in of samples, maintenance of sample documentation. Anametrix, Inc. San Jose, California, April to July 1987.

**Laboratory Technician** - Performed various tests related to soil mechanics, i.e., direct shear test, unconfined compression, compaction test, consolidation test, plasticity index, permeability test, sand equivalent, gradation, moisture and density test. Also performed maintenance of laboratory instruments and field inspections. Developed and applied skills in NMR, IR, GC (performance as well as interpretation), Mass Spectroscopy (interpretation); various purification procedures including gravity and vacuum filtration, column chromatography, and extraction. United Soil Engineering, Inc., San Jose, California, May 1983 to March 1987.

## **CHERYL BALMER - TPH SUPERVISOR**

### **EDUCATION:**

B.S. Chemistry with Minors in Biology and Sociology, Indiana University, Bloomington, 1989.

### **EXPERIENCE:**

**TPH Supervisor** - Supervision of seven chemists and data entry personnel in the analysis of petroleum hydrocarbons by gas chromatography. Responsible for project scheduling, review of reports, instrument maintenance and trouble shooting, and method development. Anametrix, Inc., San Jose, California, September 1990 to present.

**Chemist** - Analysis of soil and water samples for gasoline with BTEX and nonhalogenated volatiles. Responsible for sample tracking, calculation of results, and instrument maintenance. Anametrix, Inc., San Jose, California, August 1989 to September 1990.

## **JOSEPHINE DECARLI - SAMPLE CUSTODIAN**

### **EDUCATION:**

Diploma, Buchser High School, Santa Clara, California. 1968-71.  
Fundamentals of Chemistry (Course work), UC Santa Cruz Extension,  
Tekmart, Santa Clara, California, September-December 1989.  
Supervisory Skills Development, a training course, San Jose, California,  
November 1991.

### **EXPERIENCE / BACKGROUND:**

**Sample Receiving Supervisor** - Supervision of Sample Receiving technicians for sample login, sample storage, bottle preparation, and shipping and receiving. Development and use of application software for Sample Receiving. Anamatrix, Inc., San Jose, California, March 1992 to present.

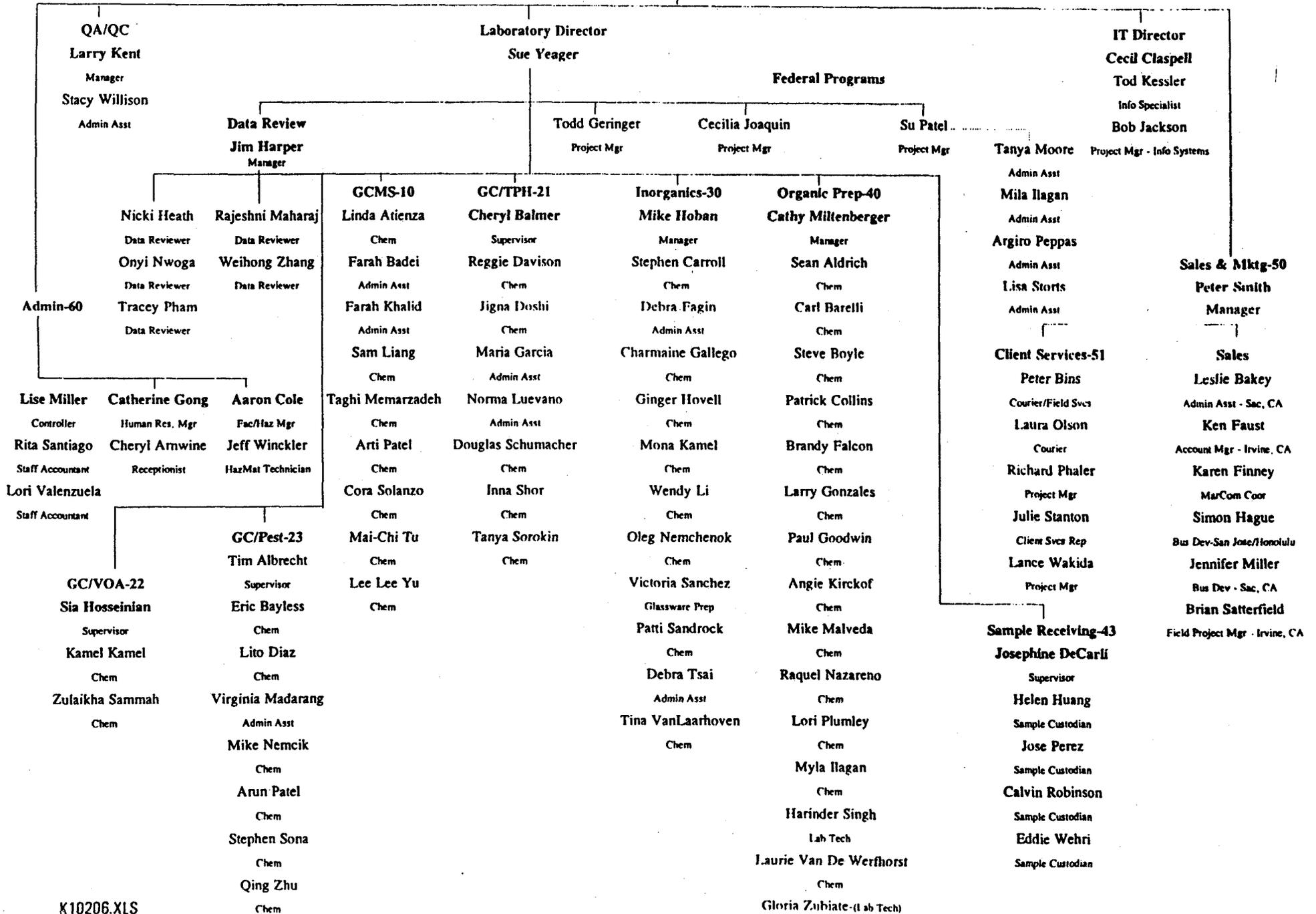
**Sample Receiving Supervisor** - Directed and supervised sample control personnel, shipping and receiving and courier services. Responsible for training new staff in sample receiving, processing all workorders and receiving them for accuracy. Acted as sample receiving custodian. IT Analytical Services, San Jose, California, June 1991 to March 1992.

**Sample Management** - In charge of sample intake, logging orders in SAM, distribution of samples to various departments and answering telephone. IT Analytical Services, San Jose, California, September 1989 to June 1991.

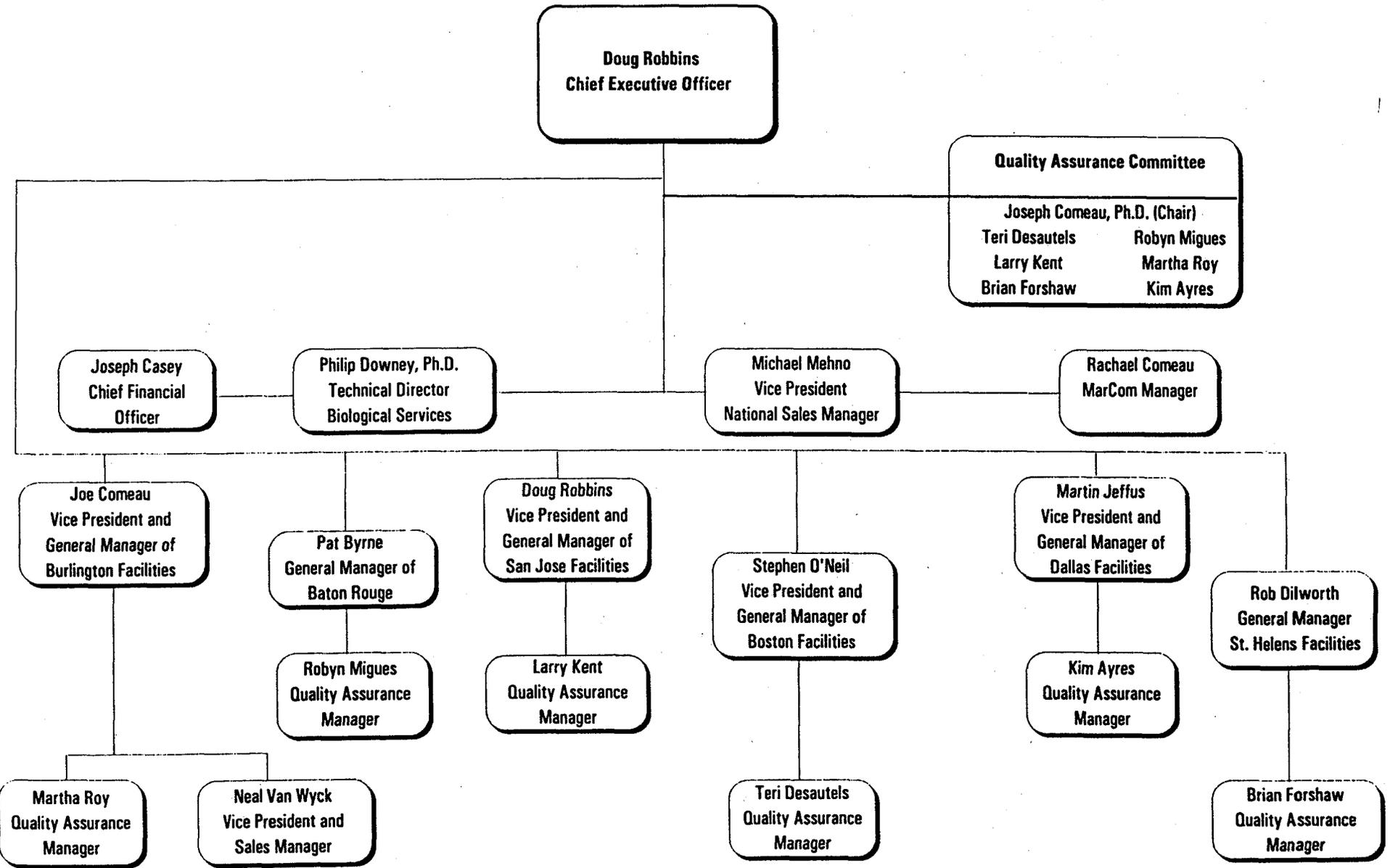
**Customer Service Representative** - Received inquiries regarding laboratory services, sample scheduling and price quotations. Performed customer satisfaction surveys and interfaced with marketing and sales staff. Coordinated incoming projects with operational staff. IT Analytical Services, San Jose, California, November 1988 to September 1989.

## **APPENDIX B**

**DOU BINS**  
**CEO**



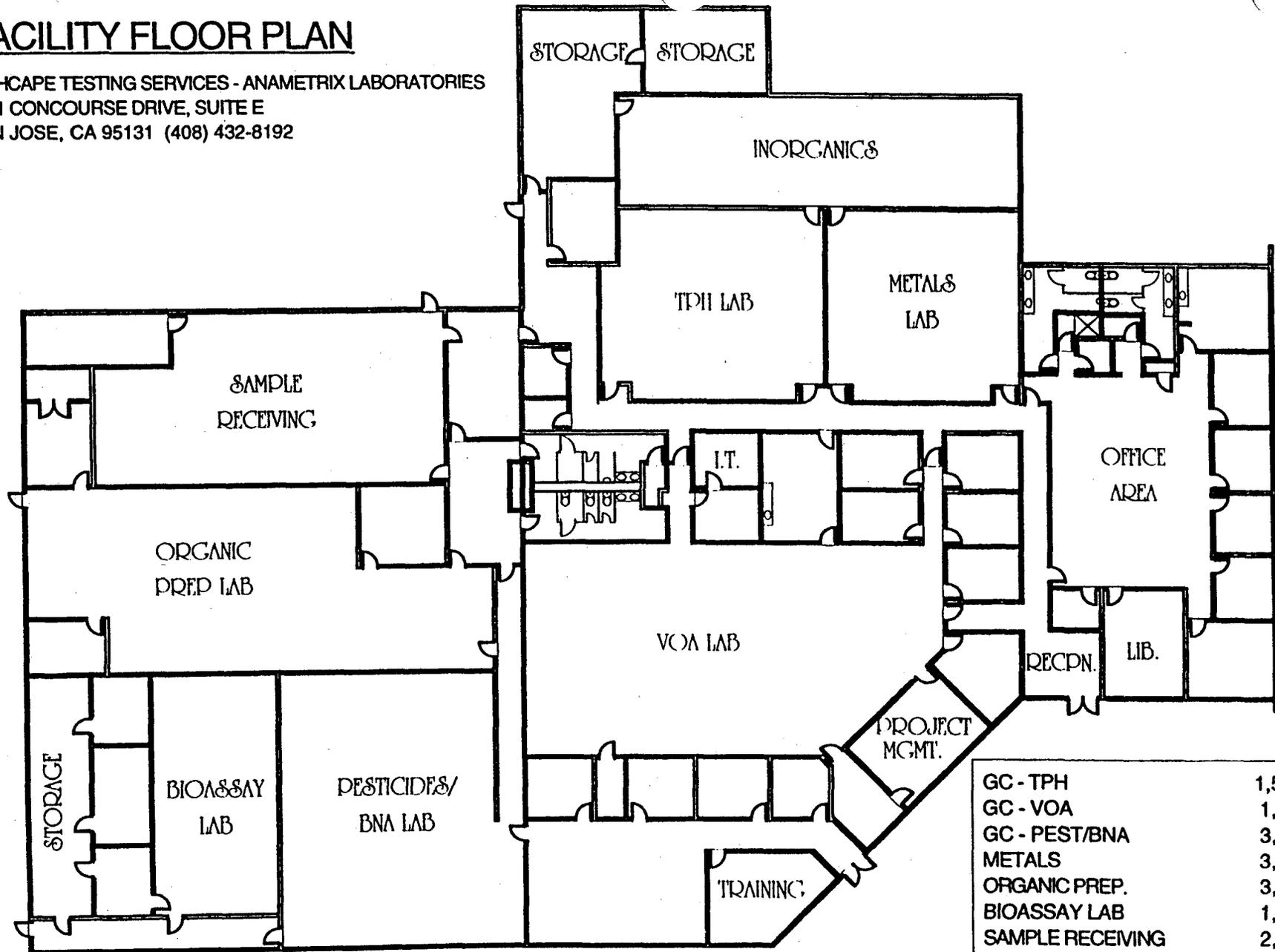
## Inchcape Testing Services/ Environmental Laboratories Management Organization Chart



**APPENDIX C**

# FACILITY FLOOR PLAN

INCHCAPE TESTING SERVICES - ANAMETRIX LABORATORIES  
 1961 CONCOURSE DRIVE, SUITE E  
 SAN JOSE, CA 95131 (408) 432-8192



GC - TPH	1,512 SQ. FT.
GC - VOA	1,690 SQ. FT.
GC - PEST/BNA	3,136 SQ. FT.
METALS	3,000 SQ. FT.
ORGANIC PREP.	3,000 SQ. FT.
BIOASSAY LAB	1,250 SQ. FT.
SAMPLE RECEIVING	2,016 SQ. FT.
INFORMATION TECHNOLOGY	300 SQ. FT.
<u>OFFICE/STORAGE AREAS</u>	<u>14,835 SQ. FT.</u>
<b>TOTAL</b>	<b>30,739 SQ. FT.</b>

## APPENDIX D - FIELD SAMPLING GUIDE

<b>FIELD SAMPLING GUIDE</b>							
<b>ORGANIC</b>	<b>Water and Waste Water</b>					<b>Soil and Sludge</b>	
Analyte	Container	Volume	Preservation	Hold Time	Sample Size	Hold Time	
Halogenated Volatile Organics (601/8010)	G-TLS	3x40ml	None; (1)	14 days	60g	14 days	
Aromatic Volatile Organics (602/8020)	G-TLS	3x40ml	HCl to pH<2; (1)	14 days	60g	14 days	
In Series (601/602 - 8021)	G-TLS	3x40ml	HCl to pH<2; (1)	14 days	60g	14 days	
Acrolein, Acrylonitrile & Acetonitrile (603/8030)	G-TLS	2x40ml	None; (1)	14 days (3)	60g	14 days	
Phenols (604/8030)	G-TLC	2x1 liter	None; (1)	7 days	120g	14 days	
Phthalate Esters (606/8060)	G-TLC	2x1 liter	None; (1)	7 days	120g	14 days	
Organochlorine Pesticides, PCBs (608/8080)	G-TLC	2x1 liter	pH 5-9	7 days	120g	14 days	
TCLP (8080)	G-TLC	2x1 liter	pH 5-9	7 days	120g	14 days	
Polynuclear Aromatic Hydrocarbons (610/8310)	G-TLC	2x1 liter	None; (1) store in dark	7 days	120g	14 days	
Nitroaromatics & Nitramines (8330)	G-TLC	2x1 liter	None; (1) store in dark	7 days	120g	14 days	
Haloethers (611/8110)	G-TLC	2x1 liter	None; (1)	7 days	120g	14 days	
Chlorinated Hydrocarbons (612/8120)	G-TLC	2x1 liter	None	7 days	120g	14 days	
Organophosphate Pesticides (614/8140)	G-TLC	2x1 liter	pH 5-9	7 days	120g	14 days	
Organophosphate Herbicides (615/8150)	G-TLC	2x1 liter	None	7 days	120g	14 days	
TCLP (8150)	G-TLC	2x1 liter	None	7 days	120g	14 days	
Volatile Organics by GC/MS (624/8240)	G-TLS	3 x40ml	HCl; pH<2	14 days	120g	14 days	
TCLP (8240)	G-TLS	3x40ml	None	14 days	100g	14 days	
Organic Lead (LUFT)	G-TLC	1 liter	None	14 days	100g	14 days	
Semivolatile Organics by GC/MS (625/8270)	G-TLC	2x1 liter	None	7 days	120g	14 days	
TCLP (8270)	G-TLC	2x1 liter	None	7 days	120g	14 days	
Carbamate and Urea Pesticides (632)	G-TLC	2x1 liter	None	7 days	120g	14 days	
Total Petroleum Hydrocarbons as Diesel	G-TLC	2x1 liter	None	14 days	120g	14 days	
Total Petroleum Hydrocarbons as Gasoline with BTEX	G-TLS	3x40ml	HCl; pH<2	14 days	60g	14 days	
Total Recoverable Petroleum Hydrocarbons by IR (418.1)	G-TLC	1 liter	HCl; pH<2	28 days	100g	28 days	
Oil & Grease (5520BF/EF)	G-TLC	2x1 liter	HCl; pH<2	28 days	100g	28 days	
EDB and DBCP (504/8011)	G-TLS	2x40ml	None	28 days	50g	28 days	

**Notes:**

G = Glass; TLS = Teflon Lined Septum; TLC = Teflon Lined Cap

(1) In the presence of residual chlorine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

(2) All hazardous waste samples must be double contained in a Mason Jar or a Ziplock bag.

(3) For Acrolein with no pH adjustment using HCl, sample must be analyzed within 3 days.

(4) Brass liner/Steel Tube; Teflon Lined Cap.

Loose sludges should be collected in jars, not tubes.

Do not allow any headspace in container for all volatile organics.

No chemical preservation should be used for organic liquids such as oils, solvents, or fuels. For such samples, please contact our Client Services Department for specific instructions.

**All Samples Must Be Kept Cool (4°C)**

GENERAL CHEMISTRY	Water and Waste Water				Soil and Sludge	
	Analyte	Container	Volume	Preservation	Hold Time	Sample Size
All Metals (6010/200.7/ 200 & 7000 series)	P	500 mL	HNO <sub>3</sub> , pH<2(1)	6 mos	100g	6 mos
TCLP Metals	P	500 mL	None	6 mos	100g	6 mos
Chromium VI (7195/7196/218)	P	500 mL	None	24 hours	50g	7 days (3)
Mercury (7470/7471/245.1/245.5)	P	500 mL	HNO <sub>3</sub> , pH<2(1)	28 days	50g	13 days
Acidity (305)	P,G	200 mL	None	14 days	50g	14 days
Alkalinity (310.1)	P,G	500 mL	None	14 days	50g	14 days
Ammonia (350.3)	P,G	500 mL	H <sub>2</sub> SO <sub>4</sub> , pH<2	28 days	50g	28 days
Biochemical Oxygen Demand (405.1)	P,G	1 liter	None	48 hours	N/A	N/A
Bromide (320.1/ 300.0)	P,G	500 mL	None	28 days	50g	28 days
Bromate (300.0)	P,G	500 mL	None	28 days	50g	28 days
Chemical Oxygen Demand (410)	P,G	200 mL	H <sub>2</sub> SO <sub>4</sub> ,pH<2	28 days	N/A	N/A
Chloride (300.0)	P,G	500 mL	None	28 days	50g	28 days
Chlorine, Total Residue (330)	P,G	500 mL	None	Immediately	N/A	N/A
Chlorate (300.0)	P,G	500 mL	None	28 days	50g	28 days
Color (110)	P,G	100 mL	None	48 hours	N/A	N/A
Chlorite (300.0)	P,G	500 mL	None	28 days	50g	28 days
Cyanide (335/9010) Total and Amenable to Chlorination	P,G	500 mL	NaOH,pH>12(2)	14 days	50g	14 days
Fluoride (340.2)	P,G	500 mL	None	28 days	50g	28 days
Formaldehyde (8315)	G-TLC	1x1 liter	None	5 days		
Hardness (2340B)	P	500ml	HNO <sub>3</sub> ,pH<2	6 mos	N/A	N/A
Ignitability (1010)	P,G	250 mL	None	6 mos	50g	6 mos
pH (150.1/9040/9045)	P,G	40 mL	None	Immediately	50g	Immed.
Nitrogen, Kjeldahl and Organic (351)	P,G	500 mL	H <sub>2</sub> SO <sub>4</sub> ,pH<2	28 days	50g	28 days
Nitrate (300.0)	P,G	500 mL	None	48 hours	50g	48 hrs
Nitrate-Nitrite (353)	P,G	200 mL	H <sub>2</sub> SO <sub>4</sub> ,pH<2	28 days	50g	28 days
Nitrite (354/300.0)	P,G	200 mL	None	48 hours	50g	48 hrs
Total Organic Carbon (415/9060)	G	200 mL	H <sub>2</sub> SO <sub>4</sub>	28 days	50g	28 days
Orthophosphate (365/300.0)	P,G	200 mL	Filter on site	48 hours	50g	48 hrs
Phenolics (420)	G	500 mL	H <sub>2</sub> SO <sub>4</sub> ,pH<2	28 days	50g	28 days
Phosphorus, Total (365)	P,G	200 mL	H <sub>2</sub> SO <sub>4</sub> ,pH<2	28 days	50g	28 days
TDS/Filterable Residue (160.1)	P,G	500 mL	None	7 days	N/A	N/A
TSS/Nonfilterable Residue (160.2)	P,G	500 mL	None	7 days	N/A	N/A
Residue, Total (160.3)	P,G	500 mL	None	7 days	N/A	N/A
Residue, Volatile (160.4)	P,G	500 mL	None	7 days	N/A	N/A
Residue, Settleable (160.5)	P,G	1000 mL	None	48 hours	N/A	N/A
Silica (370.1)	P	200 mL	None	28 days	N/A	N/A

<b>GENERAL CHEMISTRY</b>		<b>Water and Waste Water</b>			<b>Soil and Sludge</b>	
Analyte	Container	Volume	Preservation	Hold Time	Sample Size	Hold Time
Specific Conductance (120.1/9050)	P,G	100 mL	None	24 hours	50g	28 da
Sulfate (300.0)	P,G	200 mL	None	28 days	50g	28 days
Sulfide (376/9030)	P,G	500 mL	Zinc Acetate & NaOH,pH>9	7 days	50g	7 days
Sulfite (377)	P,G	200 mL	None	Immediately	50g	Immed.
Surfactants-MBAS (425.1)	P,G	1 liter	None	48 hours	N/A	N/A
Turbidity (180.1)	P,G	200 mL	None	48 hours	N/A	N/A

**Notes:**

P = Polyethylene G = Glass

(1) For dissolved metals, samples should be filtered immediately on site prior to adding preservative.

(2) In the presence of residual chlorine, add 0.6g of ascorbic acid.

(3) There is no documentation regarding hold time for this element in soil. Inchcape Testing Services will analyze as soon as possible with a maximum within 7 days.

**All samples must be kept cool (4°C)**

**DRINKING WATER**

Analyte	Container	Volume	Preservation	Hold Time
Volatile Halogenated Organics (502.1)	G-TLS	3x40ml	HCl;pH<2	14 days
Volatile Organics (502.2)	G-TLS	3x40ml	HCl;pH<2	14 days
Volatile Aromatics (503.1)	G-TLS	3x40ml	HCl;pH<2	14 days
EDB and DBCP (504)	G-TLS	2x40ml	HCl;pH<2(1)	28 days
Organohalide Pesticides and PCBs (505)	G-TLS	2x40 mL	None;(3)	14 days (2)
Chlorinated Pesticides (508)	G-TLC	2x1 liter	None;(3); (4)	7 days
PCB Screen (508A)	G-TLC	2x1 liter	None	14 days
Volatile Organics (524.2)	G-TLS	3x40ml	HCl;pH<2	14 days
Semivolatile Organics by GC/MS (525)	G-TLC	2x1 liter	HCl;pH<2;(5)	7 days
N-methylcarbamoyloximes & N-methylcarbamates (531.1)	G-TLC	2x1 liter	HCl; pH3;(3)	28 days
Polyaromatic Hydrocarbons (550)	G-TLC	2x1 liter	None	7 days
Inorganic Anions by Chromatography (300.0)	P,G-TLC	500 mL	None	Depends on each anion
Metals (200.7/200 series)	P	500 mL	HNO <sub>3</sub> to pH<2	6 mos
Chromium VI (218)	P	500 mL	None	24 hours
Mercury (245.1)	P	500 mL	HNO <sub>3</sub> to pH<2	28 days

**Notes:**

P = Polyethylene G = Glass TLS = Teflon Lined Septum TLC = Teflon Lined Cap

(1) If sample is chlorinated, add 3 mg of sodium thiosulfate.

(2) If heptachlor is of concern, samples must be extracted within 7 days, if not, then 14.

(3) If sample is chlorinated, add 80 mg of sodium thiosulfate per liter of sample.

(4) Add 1 mL of a 10 mg/mL mercuric chloride solution to each liter of sample. Use caution when handling.

(5) If sample is chlorinated, add 40-50 mg of sodium sulfite per liter. If sample is unchlorinated, adjust the pH to less than 2 with 6N HCl.

**All samples must be kept cool (4°C)**

The hold time listed is the maximum time that samples may be held before analysis and still be considered valid. Samples should be analyzed immediately after collection. Inchcape Testing Services is not responsible for the accuracy of the information contained in this document. This document is for guidance only. Users are encouraged to refer to the current regulations from which this information is obtained. For further information, contact our Quality Assurance Department at (800) 978-TEST.

References: 1) 40 CFR, Part 136.3, Table II; 2) SW-846, 1986, Test Methods for Evaluating Solid Waste

**APPENDIX E**

<b>SAMPLE RECEIVING CHECKLIST</b>			
<i>Workorder Number:</i>	<i>Client Project ID:</i>	<i>Quote Number:</i>	
<i>Cooler</i>			
Shipping documentation present? If YES, enter Carrier and Airbill #:	YES	NO	N/A
Custody Seal on the outside of cooler? <i>Condition:</i> Intact <input type="checkbox"/> Broken <input type="checkbox"/>	YES	NO	N/A
Temperature of sample(s) within range? List temperatures of cooler(s): <i>Note:</i> If all samples taken within previous 4 hr, circle N/A and place in sample storage area as soon as possible.	YES	NO	N/A
<i>Samples</i>			
Chain of custody seal present for each container? <i>Condition:</i> Intact <input type="checkbox"/> Broken <input type="checkbox"/>	YES	NO	N/A
Samples arrived within holding time?	YES	NO	N/A
Samples in proper containers for methods requested? <i>Condition of containers:</i> Intact <input type="checkbox"/> Broken <input type="checkbox"/> If NO, were samples transferred to proper container(s)? Yes <input type="checkbox"/> No <input type="checkbox"/>	YES	NO	
Were VOA containers received with zero headspace? If NO, were bubbles < 6 mm? Yes <input type="checkbox"/> No <input type="checkbox"/>	YES	NO	N/A
Were container labels complete? (ID, date, time, preservative)	YES	NO	N/A
Were samples properly preserved? If NO, was the preservative added at time of receipt? Yes <input type="checkbox"/> No <input type="checkbox"/>	YES	NO	N/A
pH check of samples required at time of receipt? If YES, pH checked and recorded by:	YES	NO	
Sufficient amount of sample received for methods requested? If NO, has the client or PM been notified? Yes <input type="checkbox"/> No <input type="checkbox"/>	YES	NO	
Field blanks received with sample batch?	YES	NO	N/A
Trip blanks received with sample batch?	YES	NO	N/A
<i>Chain of Custody</i>			
Chain of custody form received with samples?	YES	NO	
Has it been filled out completely and in ink?	YES	NO	
Sample IDs on chain of custody form agree with labels?	YES	NO	
Number of containers on chain agree with number received?	YES	NO	
Analysis methods specified?	YES	NO	
Sampling date and time indicated?	YES	NO	
Proper signatures of sampler, courier and custodian in appropriate spaces? With time and date? Yes <input type="checkbox"/> No <input type="checkbox"/>	YES	NO	
Turnaround time? Standard <input type="checkbox"/> Rush <input type="checkbox"/>			

**Any NO responses and/or any BROKEN that was checked must be detailed in a Corrective Action Form.**

Sample Custodian: \_\_\_\_\_ Date: \_\_\_\_\_ Project Manager: \_\_\_\_\_ Date: \_\_\_\_\_

**APPENDIX F**



**APPENDIX G**

**INCHCAPE TESTING SERVICES  
ENVIRONMENTAL LABORATORIES**  
1961 Concourse Drive, Suite E  
San Jose, CA 95131  
(408) 432-8192

**ANALYTICAL METHOD CHANGE REQUEST FORM**

**Date** \_\_\_\_\_ **Method No.** \_\_\_\_\_  
**Method Title:** \_\_\_\_\_ **SOP No.** \_\_\_\_\_  
**Requested By:** \_\_\_\_\_

**Change From:** \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Change To:** \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Reason for Change:** \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Approvals:**  
**Dept Supervisor:** \_\_\_\_\_ **Date:** \_\_\_\_\_

**QA Manager:** \_\_\_\_\_ **Date:** \_\_\_\_\_

## APPENDIX H

## DEPARTMENT OF HEALTH SERVICES

151 BERKELEY WAY  
BERKELEY, CA 94704-1011  
(510) 540-2800



November 16, 1994

Corinne N. Pham  
Inchcape Testing Services  
Anamatrix Laboratories  
1961 Concourse Drive, Suite E  
San Jose, CA 95131

Certificate No.: 1234

Dear Ms. Pham:

This is to advise you that the laboratory named above has been certified/ registered as an environmental testing laboratory pursuant to the provisions of the California Environmental Laboratory Improvement Act of 1988 (Health and Safety Code, Division 1, Part 2, Chapter 7.5, commencing with Section 1010).

The fields of testing for which this laboratory has been certified/registered under this Act are indicated in the enclosed "List of Approved Fields of Testing and Analytes." Certification/registration shall remain in effect until July 31, 1996 unless revoked. This certificate is subject to an annual fee as prescribed by Section 1017(a), Health and Safety Code, on the anniversary date of the certificate.

Please note that your laboratory is required to notify the Environmental Laboratory Accreditation Program of any major changes in the laboratory such as the transfer of ownership, change of laboratory director, change in location, or structural alterations which may affect adversely the quality of analyses (Section 1014(b), California Health & Safety Code).

Until the new regulations pertaining to environmental laboratories are adopted under the Act, the existing regulations pertaining to drinking water and hazardous waste testing laboratories (California Code of Regulations, Title 22, Sections 64481-64499 and 67440.1-67440.7) will remain in effect to the extent that they are not superseded by the provisions of the Act.

Your continued cooperation is essential in order to establish a reputation for the high quality of the data produced by environmental laboratories certified by the State of California.

If you have additional questions, please contact Mr. William Ray at (510) 540-2800.

Sincerely,

A handwritten signature in black ink that reads "George C. Kulasingam".

George C. Kulasingam, Ph.D., Manager  
Environmental Laboratory  
Accreditation Program

Enclosure

ENVIRONMENTAL LABORATORY ACCREDITATION/REGISTRATION  
List of Approved Fields of Testing and Analytes

Inchcape Testing Services  
Anamatrix Laboratories  
1961 Concourse Drive, Suite E  
San Jose, CA

TELEPHONE No: (408) 432-8192  
CALIFORNIA COUNTY: Santa Clara

CERTIFICATE NUMBER: 1234  
EXPIRATION DATE: 07-31-96

<b>1 Microbiology of Drinking Water and Wastewater (-----)</b>			
1.1	Total Coliforms in Drinking Water by Multiple Tube Fermentation	-----	N
1.2	Fecal Coliforms/E. Coli in Drinking Water by MTF	-----	N
1.3	Total Coliforms in Drinking Water by Membrane Filter Technics	-----	N
1.4	Fecal Coliforms/E. Coli in Drinking Water by Membrane Filter Technics	-----	N
1.5	Total Coliforms and E. Coli in Drinking Water by MMO-MUG	-----	N
1.6	Total Coliforms in Drinking Water by Clark's Presence/Absence	-----	N
1.7	Fecal Coliforms/E. Coli in Drinking Water by Clark's Presence/Absence	-----	N
1.8	Heterotrophic Plate Count	-----	N
1.9	Total Coliforms in Wastewater by Multiple Tube Fermentation	-----	N
1.10	Fecal Coliforms in Wastewater by MTF	-----	N
1.11	Total Coliforms in Wastewater by Membrane Filter Technics	-----	N
1.12	Fecal Coliforms in Wastewater by Membrane Filter Technics	-----	N
1.13	Fecal Streptococci or Enterococci by Multiple Tube Technics	-----	N
1.14	Fecal Streptococci or Enterococci by Membrane Filter Technics	-----	N
<b>2 Inorganic Chemistry and Physical Properties of Drinking Water excluding Toxic Chemical Elements (07-09-90)</b>			
2.1	Alkalinity	----- Y	
2.2	Calcium	----- Y	
2.3	Chloride	----- Y	
2.4	Corrosivity	----- N	
2.5	Fluoride	----- Y	
2.6	Hardness	----- Y	
2.7	Magnesium	----- Y	
2.8	MBAS	----- Y	
2.9	Nitrate	----- Y	
2.10	Nitrite	----- Y	
2.11	Sodium	----- Y	
2.12	Sulfate	----- Y	
2.13	Total Filterable Residue and Conductivity	----- Y	
2.14	Iron (Colorimetric Methods Only)	----- N	
2.15	Manganese (Colorimetric Methods Only)	----- N	
2.16	Phosphate, ortho	----- Y	
2.17	Silica (Colorimetric Methods Only)	----- N	
2.18	Cyanide	----- Y	
<b>3 Analysis of Toxic Chemical Elements in Drinking Water (07-09-90)</b>			
3.1	Arsenic	----- Y	
3.2	Barium	----- Y	
3.3	Cadmium	----- Y	
3.4	Chromium, total	----- Y	
3.5	Copper	----- Y	
3.6	Iron	----- Y	
3.7	Lead	----- Y	
3.8	Manganese	----- Y	
3.9	Mercury	----- Y	
3.10	Selenium	----- Y	
3.11	Silver	----- Y	
3.12	Zinc	----- Y	
3.13	Aluminum	----- Y	
3.14	Asbestos	----- N	
3.15	EPA Method 200.7	----- Y	
3.16	EPA Method 200.8 (Unregulated Elements and Lead Only)	----- N	
3.17	Antimony	----- Y	
3.18	Beryllium	----- Y	
3.19	Nickel	----- Y	
3.20	Thallium	----- Y	
<b>4 Organic Chemistry of Drinking Water (measurement by GC/MS combination) (12-30-91)</b>			
4.1	EPA Method 501.3	-----	N
4.2	EPA Method 524.2	-----	Y
4.3	EPA Method 525	-----	N
4.4	EPA Method 513	-----	N
<b>5 Organic Chemistry of Drinking Water (excluding measurements by GC/MS combination) (08-08-90)</b>			
5.1	EPA Method 501.1	----- N	
5.2	EPA Method 501.2	----- N	
5.3	EPA Method 502.1	----- N	
5.4	EPA Method 502.2	----- N	
5.5	EPA Method 503.1	----- N	
5.6	EPA Method 504	----- Y	
5.7	EPA Method 505	----- N	
5.8	EPA Method 506	----- N	
5.9	EPA Method 507	----- N	
5.10	EPA Method 508	----- N	
5.11	EPA Method 508A	----- N	
5.12	EPA Method 510.1	----- N	
5.13	EPA Method 515.1	----- N	
5.14	EPA Method 531.1	----- N	
5.15	EPA Method 547	----- N	
5.16	EPA Method 548	----- N	
5.17	EPA Method 549	----- N	
5.18	EPA Method 550	----- N	
5.19	EPA Method 550.1	----- N	
5.20	EPA Method 551	----- N	
5.21	EPA Method 552	----- N	

**6 Radiochemistry (-----)**

6.1	Gross Alpha and Beta Radiation -----	N	6.11	Gross Alpha by Co-precipitation -----	N
6.2	Total Radium -----	N	6.12	Radium 228 -----	N
6.3	Radium 226 -----	N	6.13	Radioactive Iodine -----	N
6.4	Uranium -----	N	6.14	Gross Alpha & Beta in Hazardous Wastes --	N
6.5	Radon 222 -----	N	6.15	Alpha Emitting Radium Isotopes	
6.6	Radioactive Cesium -----	N		in Haz. Wastes -----	N
6.7	Iodine 131 -----	N	6.16	Radium 228 in Hazardous Wastes -----	N
6.8	Radioactive Strontium -----	N			
6.9	Tritium -----	N			
6.10	Gamma and Photon Emitters -----	N			

**7 Shellfish Sanitation (-----)**

7.1	Shellfish meat Microbiology -----	N
7.2	Paralytic Shellfish Poison -----	N
7.3	Domoic Acid -----	N

**8 Aquatic Toxicity Bioassays (-----)**

8.1	Hazardous Waste Aquatic Toxicity Bioassay (Title 22, CCR, 66261.24(a)(6)) -----	N
8.2	Wastewater Testing According to Kopperdahl (1976) using Freshwater Fish. -----	N
8.3	Wastewater Testing According to EPA/600/4-85/013 using Freshwater and/or Marine Organisms -----	N
8.4	Wastewater Testing by EPA Method 1000.0 -----	N
8.5	Wastewater Testing by EPA Method 1002.0 -----	N
8.6	Wastewater Testing by EPA Method 1003.0 -----	N
8.7	Wastewater Testing by EPA Method 1006 -----	N
8.8	Wastewater Testing by EPA Method 1007 -----	N
8.9	Wastewater Testing by EPA Method 1009 -----	N
8.10	Wastewater Testing According to Anderson, et. al. (1990) using Giant Kelp ( <u>Macrocystis pyrifera</u> ) --	N
8.11	Wastewater Testing According to Anderson, et. al. (1990) using Red Abalone ( <u>Haliotis rufescens</u> ) ---	N
8.12	Wastewater Testing According to Dinnel and Stober (1987) using Purple Sea Urchin ( <u>Strongylocentrotus purpuratus</u> ) -----	N
8.13	Wastewater Testing According to Dinnel and Stober (1987) using Red Sea Urchin ( <u>Strongylocentrotus franciscanus</u> ) -----	N
8.14	Wastewater Testing According to Dinnel and Stober (1987) using Sand Dollar ( <u>Dendroaster excentricus</u> ) -----	N
8.15	Wastewater Testing According to procedure E 724-89 (ASTM, 1989) using Pacific Oyster ( <u>Crassostrea gigas</u> ) -----	N
8.16	Wastewater Testing According to procedure E 724-89 (ASTM, 1989) using California Bay Mussel ( <u>Mytilus edulis</u> ) -----	N
8.17	Wastewater Testing According to Standard Methods (APHA, 1989) using an alga ( <u>Skeletonema costatum</u> ) -----	N
8.18	Wastewater Testing According to EPA/600/4-90/027 using Freshwater and/or Marine Organisms -----	N

**9 Physical Properties Testing of Hazardous Waste (02-23-90)**

9.1	Ignitability by Flashpoint determination (Title 22, CCR, 66261.21) -----	Y
9.2	Corrosivity - pH determination (Title 22, CCR, 66261.22) -----	Y
9.3	Corrosivity - Corrosivity towards steel (Title 22, CCR, 66261.22) -----	N
9.4	Reactivity (Title 22, CCR, 66261.23) -----	Y

**10 Inorganic Chemistry and Toxic Chemical Elements of Hazardous Waste**

10.1	Antimony 7040(-----) -----	N	10.7	Cobalt 7200(08-25-88) -----	Y
	7041(08-25-88) -----	Y		7201(08-25-88) -----	Y
10.2	Arsenic 7060(08-25-88) -----	Y	10.8	Copper 7210(08-25-88) -----	Y
	7061(08-25-88) -----	Y		7211(08-08-90) -----	Y
10.3	Barium 7080(-----) -----	N	10.9	Lead 7420(08-25-90) -----	Y
	7081(08-25-88) -----	Y		7421(08-25-90) -----	Y
10.4	Beryllium 7090(-----) -----	N	10.10	Mercury 7470(08-25-90) -----	Y
	7091(08-25-88) -----	Y		7471(08-25-90) -----	Y
10.5	Cadmium 7130(-----) -----	N	10.11	Molybdenum 7480(-----) -----	N
	7131(08-25-88) -----	Y		7481(08-25-90) -----	Y
10.6	Chromium, total 7190(08-25-88) -----	Y	10.12	Nickel 7520(08-25-90) -----	Y
	7191(08-25-88) -----	Y			

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10.13	Selenium				
	7740(08-25-90)	-----	Y		
	7741(08-25-90)	-----	Y		
10.14	Silver				
	7760(-----)	-----	N		
	7761(08-08-90)	-----	Y		
10.15	Thallium				
	7840(-----)	-----	N		
	7841(08-25-88)	-----	Y		
10.16	Vanadium				
	7910(-----)	-----	N		
	7911(08-25-88)	-----	Y		
10.17	Zinc				
	7950(08-25-88)	-----	Y		
	7951(08-08-90)	-----	Y		
10.18	Chromium (VI)				
	7195(08-25-88)	-----	Y		
	7196(08-25-88)	-----	Y		
	7197(08-25-88)	-----	Y		
	7198(-----)	-----	N		
10.19	Cyanide				
	9010(02-23-90)	-----	Y		
10.20	Fluoride				
	300.0(06-21-90)	-----	Y		
	340.1(-----)	-----	N		
	340.2(09-19-89)	-----	Y		
	340.3(-----)	-----	N		
10.21	Sulfide				
	9030(02-23-90)	-----	Y		
10.22	Total Organic Lead				
	(08-25-88)	-----	Y		
10.23	EPA Method 6010(11-15-88)	-----	Y		
10.24	EPA Method 6020(-----)	-----	N		

**11 Extraction Tests of Hazardous Waste (08-25-88)**

11.1	California Waste Extraction Test (WET) (Title 22, CCR, 66261.100, Appendix II)	-----	Y		
11.2	Extraction Procedure Toxicity	-----	Y		
11.3	Toxicity Characteristic Leaching Procedure (TCLP) All Classes	-----	Y		
11.4	Toxicity Characteristic Leaching Procedure (TCLP) Inorganics Only	-----	N		
11.5	Toxicity Characteristic Leaching Procedure (TCLP) Extractables Only	-----	N		
11.6	Toxicity Characteristic Leaching Procedure (TCLP) Volatiles Only	-----	N		

**12 Organic Chemistry of Hazardous Waste (measurement by GC/MS combination)**

12.1	EPA Method 8240(08-09-86)	-----	Y		
12.2	EPA Method 8250(-----)	-----	N		
12.3	EPA method 8270(08-09-86)	-----	Y		
12.4	EPA Method 8280(-----)	-----	N		
12.5	EPA Method 8290(-----)	-----	N		
12.6	EPA Method 8260(09-10-92)	-----	Y		

**13 Organic Chemistry of Hazardous Waste (excluding measurements by GC/MS combination)**

13.1	EPA Method 8010(01-01-87)	-----	Y		
13.2	EPA Method 8015(01-01-87)	-----	Y		
13.3	EPA Method 8020(01-01-87)	-----	Y		
13.4	EPA Method 8030(-----)	-----	N		
13.5	EPA Method 8040(02-23-90)	-----	Y		
13.6	EPA Method 8060(-----)	-----	N		
13.7	EPA Method 8080(01-01-87)	-----	Y		
13.8	EPA Method 8090(-----)	-----	N		
13.9	EPA Method 8100(-----)	-----	N		
13.10	EPA Method 8120(04-20-89)	-----	Y		
13.11	EPA Method 8140(-----)	-----	N		
13.12	EPA Method 8150(-----)	-----	N		
13.13	EPA Method 8310(08-08-90)	-----	Y		
13.14	EPA Method 632 (07-09-90)	-----	Y		
13.15	Total Petroleum Hydrocarbons (LUFT Manual) (06-08-88)	-----	Y		
13.16	EPA Method 8011(09-10-92)	-----	Y		
13.17	EPA Method 8021(09-10-92)	-----	Y		
13.18	EPA Method 8070(-----)	-----	N		
13.19	EPA Method 8110(-----)	-----	N		
13.20	EPA Method 8141(09-10-92)	-----	Y		
13.21	EPA Method 8330(-----)	-----	N		

**14 Bulk Asbestos Analysis (-----)**

14.1	1% or Greater Asbestos Concentrations (Title 22, CCR, 66261.24(a)(2)(A))	-----	N		
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**15 Substances Regulated Under the California Safe Drinking Water and Toxic Enforcement Act (Proposition 65) and Not Included in Other listed Groups.**

**16 Wastewater Inorganic Chemistry, Nutrients and Demand (07-09-90)**

16.1	Acidity	-----	Y		
16.2	Alkalinity	-----	Y		
16.3	Ammonia	-----	Y		
16.4	Biochemical Oxygen Demand	-----	N		
16.5	Boron	-----	Y		
16.6	Bromide	-----	Y		
16.7	Calcium	-----	Y		
16.8	cBOD	-----	N		
16.9	Chemical Oxygen Demand	-----	Y		
16.10	Chloride	-----	Y		
16.11	Chlorine Residual, total	-----	Y		
16.12	Cyanide	-----	Y		
16.13	Cyanide amenable to Chlorination	-----	Y		
16.14	Fluoride	-----	Y		
16.15	Hardness	-----	Y		
16.16	Kjeldahl Nitrogen	-----	N		
16.17	Magnesium	-----	Y		
16.18	Nitrate	-----	Y		
16.19	Nitrite	-----	Y		
16.20	Oil and Grease	-----	Y		
16.21	Organic Carbon	-----	N		
16.22	Oxygen, Dissolved	-----	N		

16.23	pH	Y	16.39	Surfactants (MBAS)	Y
16.24	Phenols	N	16.40	Tannin and Lignin	N
16.25	Phosphate, ortho-	Y	16.41	Turbidity	Y
16.26	Phosphorus, total	N	16.42	Iron (Colorimetric Only)	N
16.27	Potassium	Y	16.43	Manganese (Colorimetric Only)	N
16.28	Residue, Total	Y	16.44	Total Recoverable	
16.29	Residue, Filterable (TDS)	Y		Petroleum Hydrocarbons	Y
16.30	Residue, Nonfilterable (TSS)	Y	16.45	Total Organic Halides	N
16.31	Residue, Settleable (SS)	Y			
16.32	Residue, Volatile	Y			
16.33	Silica	Y			
16.34	Sodium	Y			
16.35	Specific Conductance	Y			
16.36	Sulfate	Y			
16.37	Sulfide (includes total & soluble)	Y			
16.38	Sulfite	Y			

**17 Toxic Chemical Elements in Wastewater (07-09-90)**

17.1	Aluminum	Y	17.18	Nickel	Y
17.2	Antimony	Y	17.19	Osmium	N
17.3	Arsenic	Y	17.20	Palladium	N
17.4	Barium	Y	17.21	Platinum	N
17.5	Beryllium	Y	17.22	Rhodium	N
17.6	Cadmium	Y	17.23	Ruthenium	N
17.7	Chromium (VI)	Y	17.24	Selenium	Y
17.8	Chromium, total	Y	17.25	Silver	Y
17.9	Cobalt	Y	17.26	Strontium	N
17.10	Copper	Y	17.27	Thallium	Y
17.11	Gold	N	17.28	Tin	N
17.12	Iridium	N	17.29	Titanium	N
17.13	Iron	Y	17.30	Vanadium	Y
17.14	Lead	Y	17.31	Zinc	Y
17.15	Manganese	Y	17.32	EPA Method 200.7	Y
17.16	Mercury	Y	17.33	EPA Method 200.8	N
17.17	Molybdenum	Y	17.34	DCP	N
			17.35	Asbestos	N

**18 Organic Chemistry of Wastewater (measurements by GC/MS combination (07-09-90))**

18.1	EPA Method 624	Y
18.2	EPA Method 625	Y
18.3	EPA Method 1613	N
18.4	EPA Method 1625	N
18.5	EPA Method 613	N

**19 Organic Chemistry of Wastewater (excluding measurements by GC/MS combination) (07-09-90-)**

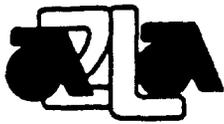
19.1	EPA Method 601	Y	19.8	EPA Method 608	Y
19.2	EPA Method 602	Y	19.9	EPA Method 609	N
19.3	EPA Method 603	N	19.10	EPA Method 610	Y
19.4	EPA Method 604	Y	19.11	EPA Method 611	N
19.5	EPA Method 605	N	19.12	EPA Method 632	Y
19.6	EPA Method 606	N	19.13	EPA Method 619	Y
19.7	EPA Method 607	N	19.99	EPA Method 614	Y

**20 Inorganic Chemistry and Toxic Chemical Elements of Pesticide Residues in Food (-----)**

20.1	Processed Foods by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetry	N
20.2	Raw Commodities by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetric	N
20.3	Dairy Products by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetry	N

EXPIRATION DATE: 07-31-96

20.4	Feed Products by One of the Following Methods	
	Atomic Absorption Spectrophotometry	N
	Inductively Coupled Plasma Atomic Emission Spectrophotometry	N
	Inductively Coupled Plasma/Mass Spectrometry	N
	Colorimetry	N
21	<u>Organic Chemistry of Pesticide Residues in Food (measurements by GC/MS) (-----)</u>	
21.1	Gas Chromatographic/Mass Spectrometric Methods in Processed Foods	N
21.2	Gas Chromatographic/Mass Spectrometric Methods in Raw Commodities	N
21.3	Gas Chromatographic/Mass Spectrometric Methods in Dairy Products	N
21.4	Gas Chromatographic/Mass Spectrometric Methods in Feed Products	N
22	<u>Organic Chemistry of Pesticide Residues in Food (Excluding Measurement by GC/MS Combination) (-----)</u>	
22.1	Halogenated Compounds in Processed Foods by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.2	Organophosphorous Compounds in Processed Foods by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.3	Carbamates in Processed Foods by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.4	Halogenated Compounds in Raw Commodities by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.5	Organophosphorous Compounds in Raw Commodities by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.6	Carbamates in Raw Commodities by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.7	Halogenated Compounds in Dairy Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.8	Organophosphorous Compounds in Dairy Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.9	Carbamates in Dairy Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.10	Halogenated Compounds in Feed Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.11	Organophosphorous Compounds in Feed Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N
22.12	Carbamates in Feed Products by One of the Following Methods	
	Gas Chromatography	N
	High Pressure Liquid Chromatography	N
	Liquid Chromatography/Mass Spectrometry	N



# American Association for Laboratory Accreditation

## SCOPE OF ACCREDITATION

INCHCAPE TESTING SERVICES - ANAMETRIX LABORATORIES

1961 Concourse Drive Suite E

San Jose, CA 95131

Larry Kent Phone: 408 432 8192

### ENVIRONMENTAL

Valid To: March 31, 1996

Certificate Number: 0268-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

#### Testing Technologies

Atomic Absorption/ICP-AES Spectrometry, Gas Chromatography, Gas Chromatography/Mass Spectrometry, Gravimetry, High Performance Liquid Chromatography, Ion Chromatography, Misc.- Electronic Probes (pH, F<sup>-</sup>), Hazardous Waste Characteristics Tests, Spectrophotometry (Visible), Titrimetry

Potable Water: metals, nutrients, classical (wet) chemistry

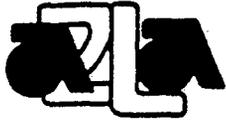
Nonpotable Water: metals, nutrients, classical (wet) chemistry, purgeable organics, extractable organics and pesticides-PCBs

Solid/Hazardous Waste: metals, nutrients, classical (wet) chemistry, purgeable organics, extractable organics, pesticides-PCBs, and hazardous waste characteristics (TCLP)

A supplemental scope, identifying the full range of tests and types of tests, is available from A2LA or the laboratory.

revised 10/09/95





# American Association for Laboratory Accreditation

## SUPPLEMENT TO SCOPE OF ACCREDITATION

INCHCAPE TESTING SERVICES - ANAMETRIX LABORATORIES  
1961 Concourse Drive Suite E  
San Jose, CA 95131  
Larry Kent Phone: 408 432 8192

### ENVIRONMENTAL

Valid as of: October 9, 1995  
Valid until: March 31, 1996

Certificate Number: 0268-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform recognized EPA methods for the following determinations:

#### Potable Water

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Au, Fe, Pb, Mg, Mn, Hg, Mo, Pd, Pl, Pt, K, Se, Si, Ag, Na, Sn, Ti, Tl, V, Zn

per EPA test methods 200.7, 204.2, 206.2, 213.2, 218.2, 220.2, 239.2, 245.2, 249.2, 270.2, 272.2, 279.2

Nutrients: Ammonia (as N), Nitrate (as N), Nitrite (as N), Orthophosphate (as P)

per EPA test methods 200.7, 300.0, 350.3

Classical Chemistry: Alkalinity, Cyanide, Fluoride, pH, Total residue, Filterable residue, Nonfilterable residue, Sulfide, Specific conductance

per EPA test methods 120.1, 150.1, 160.1, 160.2, 160.3, 310.1, 335.2, 340.2, 376.1, 376.2

#### Nonpotable Water

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Au, Fe, Pb, Mg, Mn, Hg, Mo, Pd, Pl, Pt, K, Se, Si, Ag, Na, Sn, Ti, Tl, V, Zn

per EPA test methods 6010, 7060, 7131, 7191, 7211, 7421, 7470, 7521, 7740, 7761, 7841

Nutrients: Ammonia (as N), Nitrate (as N), Nitrite (as N), Orthophosphate (as P)

per EPA test methods 300.0, 350.3, 6010

*PLM*

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Classical Chemistry: Fluoride, pH, Total residue, Filterable residue,  
Nonfilterable residue, Specific conductance, Sulfide

per EPA test methods 160.1, 160.2, 160.3, 340.2, 376.1, 376.2, 413.1, 9040,  
9050

Purgeable Organics: Acetone, Acetonitrile, Acrolein, Acrylonitrile, Benzene,  
Bromodichloromethane, Bromoform, Bromomethane, 2-Butanone, Carbon disulfide,  
Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroethylvinyl ether,  
Chloroform, Chloromethane, Dibromochloromethane, 1,2-Dichlorobenzene,  
1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane,  
1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene,  
cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane,  
cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Ethylbenzene, 2-Hexanone,  
Methylene Chloride, 4-Methyl-2-pentanone, Styrene, 1,1,1,2-Tetrachloroethane,  
1,1,2,2-Tetrachloroethane, Tetrachloroethene, Toluene, 1,1,1-Trichloroethane,  
1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, Vinyl acetate,  
Vinyl chloride, Xylene total

per EPA test methods 601, 602, 624

Extractable Organics: Acenaphthene, Acenaphthylene, Aniline, Anthracene,  
Benzidine, Benzoic acid, Benzo(a)anthracene, Benzo(b)fluoranthene,  
Benzo(k)fluoranthene, Benzo(ghi)perylene, Benzo(a)pyrene, Benzyl alcohol,  
Bis(2-chloroethoxy)methane, Bis(2-chloroethyl)ether,  
Bis(2-chloroisopropyl)ether, Bis(2-ethylhexyl)phthalate, 4-Bromophenylphenyl  
ether, Butyl benzyl phthalate, 4-Chloroaniline, 4-Chloro-3-methylphenol,  
2-Chloronaphthalene, 2-Chlorophenol, 4-Chlorophenylphenyl ether, Chrysene,  
Cresols (methyl phenols), Dibenzofuran, Dibenzo(a,e)pyrene,  
1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene,  
3,3'-Dichlorobenzidine, 2,4-Dichlorophenol, 2,6-Dichlorophenol,  
Diethylphthalate, 2,4-Dimethylphenol, Dimethylphthalate,  
Di-n-butylphthalate, Di-n-octylphthalate, 2,4-Dinitrophenol, 2,4-Dinitrotoluene,  
2,6-Dinitrotoluene, Fluoranthene, Fluorene, Hexachlorobenzene,  
Hexachlorobutadiene, Hexachlorocyclohexane, Hexachlorocyclopentadiene,  
Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone,  
2-Methyl-4,6-Dinitrophenol, 2-Methylnaphthalene, 2-Methylphenol, 4-Methylphenol,  
Naphthalene, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline, Nitrobenzene,  
2-Nitrophenol, 4-Nitrophenol, N-Nitrosodimethylamine, N-Nitrosodi-n-propylamine,  
N-Nitrosodiphenylamine, Pentachlorohexane, Pentachlorophenol, Phenacetin,  
2-Picoline, Styrene, 2,3,4,5-Tetrachlorophenol, 2,4,6-Tribromophenol,  
1,2,4-Trichlorobenzene, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol

per EPA test methods 604, 610, 612, 625

Pesticides-PCBs: Aldrin, Azinphos methyl, alpha-BHC, beta-BHC, delta-BHC,  
gamma-BHC (Lindane), Bolstar, Chlordane (technical), Chlorpyrifos, Coumaphos,  
4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Demeton-O, Demeton-S, Diazinon, Dichlorvos,  
Dieldrin, Dinoseb, Disulfoton, Endosulfan I (alpha), Endosulfan II (beta),  
Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Ethoprop,  
Fensulfothion, Fenthion, Heptachlor, Heptachlor epoxide, Malathion, Merphos,  
Methoxychlor, Mevinphos, Parathion ethyl, Parathion methyl, PCB-1016 (arochlor),  
PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260, Phorate, Propoxur,

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Ronnel, Stirophos (Tetrachlorvinphos), Tokuthion (Prothiofos), Toxaphene, Trichloronate

per EPA test methods 608, 614

Solid Waste/Hazardous Waste

Metals: Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Au, Fe, Pb, Mg, Mn, Hg, Mo, Pd, Pl, Pt, K, Se, Si, Ag, Na, Sn, Ti, Tl, V, Zn

per EPA test methods 6010, 7060, 7131, 7191, 7211, 7421, 7470, 7521, 7740, 7761, 7841

Nutrients: Ammonia (as N), Nitrate (as N), Nitrite (as N), Orthophosphate (as P)

per EPA test methods 300.0, 350.3, 6010

Classical Chemistry: Cyanide, Fluoride, pH

per EPA test methods 340.2, 9010, 9045

Purgeable Organics: Acetone, Acetonitrile, Acrylonitrile, Benzene, Bromobenzene, Bromodichloromethane, Bromoform, Bromomethane, 2-Butanone, n-Butylbenzene, sec-Butylbenzene, tert-Butylbenzene, Carbon disulfide, Carbon tetrachloride, Chlorobenzene, Chloroethane, 2-Chloroethylvinyl ether, Chloroform, Chloromethane, Dibromochloromethane, 1,2-Dibromo-3-chloropropane (DBCP), 1,2-Dibromoethane (EDB), 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, Dichlorodifluoromethane, 1,1-Dichloroethane, 1,2-Dichloroethane, 1,1-Dichloroethene, cis-1,2-Dichloroethene, trans-1,2-Dichloroethene, 1,2-Dichloropropane, 1,3-Dichloropropane, 2,2-Dichloropropane, 1,1-Dichloropropene, cis-1,3-Dichloropropene, trans-1,3-Dichloropropene, Diethyl ether, Ethanol, Ethylbenzene, 2-Hexanone, Hexachlorobutadiene, Isopropylbenzene, 1,4-Isopropyltoluene, Methylene Chloride, Methyl ethyl ketone, 4-Methyl-2-pentanone, Naphthalene, n-Propylbenzene, Styrene, 1,1,1,2-Tetrachloroethane, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, Toluene, 1,1,1-Trichloroethane, 1,1,2-Trichloroethane, Trichloroethene, Trichlorofluoromethane, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, Vinyl acetate, Vinyl chloride, Xylene total

per EPA test methods 8010, 8011, 8015, 8020, 8030, 8240, 8260.

Extractable Organics: Acenaphthene, Acenaphthylene, Aniline, Anthracene, Benzidine, Benzoic acid, Benzo(a)anthracene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Benzo(ghi)perylene, Benzo(a)pyrene, Benzyl alcohol, Bis(2-chloroethoxy)methane, Bis(2-chloroethyl)ether, Bis(2-chloroisopropyl)ether, Bis(2-ethylhexyl)phthalate, 4-Bromophenylphenyl ether, Butyl benzyl phthalate, 4-Chloroaniline, 4-Chloro-3-methylphenol, 2-Chloronaphthalene, 2-Chlorophenol, 4-Chlorophenylphenyl ether, Chrysene, Cresols (methyl phenols), Dibenzofuran, Dibenzo(a,e)pyrene, 1,2-Dichlorobenzene, 1,3-Dichlorobenzene, 1,4-Dichlorobenzene, 3,3'-Dichlorobenzidine, 2,4-Dichlorophenol, Diethylphthalate, 2,4-Dimethylphenol, Dimethylphthalate, Di-n-butylphthalate.

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Di-n-octylphthalate, 2,4-Dinitrophenol, 2,4-Dinitrotoluene,  
2,6-Dinitrotoluene, Fluoranthene, Fluorene, Hexachlorobenzene,  
Hexachlorobutadiene, Hexachlorocyclohexane, Hexachlorocyclopentadiene,  
Hexachloroethane, Indeno(1,2,3-cd)pyrene, Isophorone,  
2-Methyl-4,6-Dinitrophenol, 2-Methylnaphthalene, 2-Methylphenol,  
4-Methylphenol, Naphthalene, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline,  
Nitrobenzene, 2-Nitrophenol, 4-Nitrophenol, N-Nitrosodimethylamine,  
N-Nitrosodi-n-propylamine, N-Nitrosodiphenylamine, Pentachlorohexane,  
Pentachlorophenol, Phenacetin, 2-Picoline, Styrene, 2,3,4,5-Tetrachlorophenol,  
2,4,6-Tribromophenol, 1,2,4-Trichlorobenzene, 2,4,5-Trichlorophenol,  
2,4,6-Trichlorophenol

per EPA test methods 8040, 8120, 8270, 8310

Pesticides-PCBs: Aldrin, Azinphos methyl, alpha-BHC, beta-BHC, delta-BHC,  
gamma-BHC (Lindane), Bolstar, Chlordane (technical), Chlorpyrifos, Coumaphos,  
4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Demeton-O, Demeton-S, Diazinon, Dichlorvos,  
Dieldrin, Dinoseb, Disulfoton, Endosulfan I (alpha), Endosulfan II (beta),  
Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Ethoprop,  
Fensulfothion, Fenthion, Heptachlor, Heptachlor epoxide, Malathion, Merphos,  
Methoxychlor, Mevinphos, Parathion ethyl, Parathion methyl, PCB-1016 (arochlor),  
PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1254, PCB-1260, Phorate, Propoxur,  
Ronnel, Stirophos (Tetrachlorvinphos), Tokuthion (Prothiofos), Toxaphene,  
Trichloronate

per EPA test methods 8080, 8140

Hazardous Waste Characteristics: TCLP

per EPA test methods 1311, 1312





REPLY TO  
ATTENTION OF

DEPARTMENT OF THE ARMY  
U.S. ARMY CORPS OF ENGINEERS — MRD  
HTRW MANDATORY CENTER OF EXPERTISE  
12565 WEST CENTER ROAD  
OMAHA, NEBRASKA 68144-3869



May 2, 1996

Hazardous, Toxic and Radioactive Waste  
Center of Expertise

Inchcape Testing Services  
1961 Concourse Drive, Suite E  
San Jose, CA 95131

Gentlemen:

This correspondence addresses the recent evaluation of your laboratory by the U.S. Army Corps of Engineers (USACE) for chemical analysis in support of the USACE Hazardous Toxic and Radioactive Waste Program.

Inchcape Testing Services of San Jose, CA has successfully analyzed audit samples as listed below:

<u>METHOD</u>	<u>PARAMETERS</u>	<u>MATRIX</u>
8021	Halogenated Volatile Organics	Water <sup>(3)</sup>
8021	Halogenated Volatile Organics	Solids
8021	Aromatic Volatile Organics	Water <sup>(3)</sup>
8021	Aromatic Volatile Organics	Solids
8270A	Semivolatile Organics	Water <sup>(3)</sup>
8081	Organochlorine Pesticides	Water <sup>(3)</sup>
8081	Organochlorine Pesticides	Solids
8081	Polychlorinated Biphenyls	Water <sup>(3)</sup>
8081	Polychlorinated Biphenyls	Solids <sup>(3)</sup>
SW-846	TAL Metals <sup>(1)</sup>	Water <sup>(3)</sup>
SW-846	TAL Metals <sup>(1)</sup>	Solids <sup>(3)</sup>
9060	Total Organic Carbon	Water <sup>(3)</sup>
300 series	Anions <sup>(2)</sup>	Water <sup>(3)</sup>
Mod 8015	Total Petroleum Hydrocarbons	Water
Mod 8015	Total Petroleum Hydrocarbons	Solids

- Remarks:
- 1) TAL Metals: Aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc.
  - 2) Anions: Chloride, fluoride, sulfate, nitrate, nitrite, ortho-phosphate.
  - 3) The laboratory has successfully analyzed a performance evaluation sample for this method/matrix.
  - 4) 'Solids' includes soils, sediments, and solid waste.

Based on the successful analysis of the audit samples indicated in the table in Paragraph 2 above, Inchcape Testing Services of San Jose, CA is validated for sample analysis by the methods listed above. A full validation of 18 months was approved by the USACE Contract Laboratory Evaluation Committee on May 1, 1996.

The expiration date of validation is November 1, 1997. The Chemistry Branch of the Hazardous, Toxic, and Radioactive Waste Center of Expertise may schedule and conduct an on-site audit at any time during the 18 month validation period to evaluate lab performance if deemed necessary. USACE reserves the right to suspend validation status for any or all of the listed parameters if deemed necessary. It should be noted that your laboratory may not subcontract USACE analytical work to any other laboratory location without the approval of this office. This laboratory validation does not guarantee the delivery of any samples from a USACE Contracting Officer.

If you have any questions or comments, please contact Ms. Elena Webster at (402) 697-2574.

Sincerely,

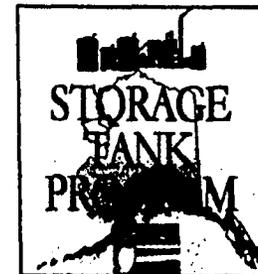


Marcia C. Davies, Ph.D.  
Director, USACE Hazardous,  
Toxic and Radioactive Waste  
Center of Expertise



# STATE OF ALASKA

DEPARTMENT OF  
ENVIRONMENTAL CONSERVATION  
**Storage Tank Program**



## *Laboratory Approval*

**Laboratory:** Inchcape Testing Services  
1961 Concourse Drive  
San Jose, California 95131

**Issued:** April 22, 1996

**Approval Number:** UST-015

**Expires:** January 11 1997

**Approved for:** Gasoline Range Organics in solids; Diesel Range Organics in waters and in solids; Residual Range Organics in solids; PAHs in waters and in solids; VCSs in waters and in solids; PCBs in waters and in solids; and Metals (Arsenic, Cadmium, Chromium and Lead) in waters and in solids

  
\_\_\_\_\_  
Donald E. LaFara  
Quality Assurance Manager

4/23/96  
Date

  
\_\_\_\_\_  
Mary Jane F. Pilgrim, Ph.D.  
Approval Authority

4/23/96  
Date

Arizona  
Department of  
Health Services

## ENVIRONMENTAL LABORATORY LICENSE

***INCHCAPE TESTING SERVICES AZ0433***

is in compliance with Environmental Laboratory's applicable standards for the  
State of Arizona and maintains on file a List of Parameters for which it is  
certified to perform analysis.

**PERIOD OF LICENSURE: FROM September 13, 1995 TO September 13, 1996**

*Wynand H. Nimmo*

Wynand H. Nimmo  
Program Manager  
State Laboratory Services



*Jack Dillenberg*

Jack Dillenberg, D.D.S., M.P.H.  
Director  
Department of Health Services

ARIZONA DEPARTMENT OF HEALTH SERVICES  
 DIVISION OF STATE LABORATORY SERVICES  
 OFFICE OF LABORATORY LICENSURE AND CERTIFICATION

Permitting Number: AZ0433

Date: February 8, 1996

Facility: INCHCAPE TESTING SERVICES

Director: SUSAN KRASKA YEAGER

Address: 1961 CONCOURSE DRIVE, SUITE E

City: SAN JOSE

County: SANTA CLARA

State: CA

Zip Code: 95131

Telephone: (408) 432-8192

Ext:

\*\*\* LIST OF LICENSED PARAMETERS AND APPROVED METHODS BY PROGRAM \*\*\*

BILLING NUMBER	PROGRAM	METHOD STATUS	PARAMETER	APPROVED METHOD
AZ0433			ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPEC. #2
			ATOMIC ABSORPTION SPECTROPHOTOMETER	AA SPECTROPH
			GAS CHROMATOGRAPH	GAS CHRO. #3
			GAS CHROMATOGRAPH	GAS CHRO. #4
			GAS CHROMATOGRAPH	GAS CHRO. #2
			GAS CHROMATOGRAPH	GAS CHROMATO
			GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS #2
			GAS CHROMATOGRAPH/MASS SPECTROMETER	GC/MS
			INDUCTIVELY COUPLED PLASMA SPECTROMETER	ICP SPECTROPH
AZ0433	HW		ALUMINUM	EPA 6010
			ANTIMONY	EPA 6010
			AROMATIC VOLATILES	EPA 8020
			ARSENIC	EPA 7060
			ARSENIC	EPA 6010
			BARIUM	EPA 6010
			BERYLLIUM	EPA 6010
			CADMIUM	EPA 6010
			CALCIUM	EPA 6010
			CHROMIUM TOTAL	EPA 6010
			COBALT	EPA 6010
			COPPER	EPA 6010
			CYANIDE TOTAL AND AMENABLE	EPA 9010
			FUEL CLASS HYDROCARBONS	BLS-191
			HALOGENATED VOLATILES	EPA 8010
			IRON	EPA 6010
			LEAD	EPA 7421
			LEAD	EPA 6010
			MAGNESIUM	EPA 6010

PROGRAMS:

AIR=AIR, HW=HAZARDOUS WASTE

SAFE DRINKING WATER, WW= WASTEWATER

STATUS:

P=PROVISIONAL

T=TEMP. LICENSE

BILLING NUMBER	PROGRAM	METHOD STATUS	PARAMETER	APPROVED METHOD
AZ0433	HW		MANGANESE	EPA 6010
			MERCURY	EPA
			MERCURY	EPA
			MOLYBDENUM	EPA 6010
			NICKEL	EPA 6010
			OIL AND GREASE	EPA 907C
			OIL AND GREASE	EPA 9071
			ORGANOCHLORINE PESTICIDES	EPA 8080
			POTASSIUM	EPA 6010
			SELENIUM	EPA 7740
			SELENIUM	EPA 6010
			SEMIVOLATILE COMPOUNDS	EPA 8270
			SILVER	EPA 6010
			SODIUM	EPA 6010
			STRONTIUM	EPA 6010
			THALLIUM	EPA 7841
			THALLIUM	EPA 6010
			TOTAL PETROLEUM HYDROCARBONS	BLS-181
			TOXICITY CHARACTERISTIC LEACHING PROCEDURE	EPA 1311
			VANADIUM	EPA 6010
			VOLATILE COMPOUNDS	EPA 8260
			VOLATILE COMPOUNDS	EPA 8240
			VOLATILE COMPOUNDS	EPA 8021
			ZINC	EPA 6010

AZ0433	SDW		ARSENIC	EPA 206.7
			BARIUM	EPA 200
			CADMIUM	EPA 200
			CALCIUM	EPA 200.7
			CHROMIUM TOTAL	EPA 200
			COPPER	EPA 200
			IRON	EPA 200.7
			LEAD	EPA 239.7
			LEAD	EPA 200
			MAGNESIUM	EPA 200
			MANGANESE	EPA 200.7
			MERCURY	EPA 245
			MOLYBDENUM	EPA 200
			NICKEL	EPA 200.7
			NICKEL	6010
			SELENIUM	EPA 270
			SILVER	EPA 200.7
			SODIUM	EPA 200.7
			STRONTIUM	EPA 200
			THALLIUM	EPA 279

PROGRAMS:  
 AIR=AIR, HW=HAZARDOUS WASTE  
 SDW=SAFE DRINKING WATER, WW= WASTEWATER

STATUS:  
 P=PROVISIONAL  
 T=TEMP. LICENSE

BILLING NUMBER	PROGRAM	METHOD STATUS	PARAMETER	APPROVED METHOD
AZ0433	SDW		VOLATILE ORGANICS ZINC	EPA 524.2 EPA 200.7
AZ0433	WW		ALUMINUM ANTIMONY AROMATIC VOLATILES ARSENIC ARSENIC BARIUM BERYLLIUM CADMIUM CALCIUM CHROMIUM TOTAL COBALT COPPER HALOGENATED VOLATILES IRON LEAD LEAD MAGNESIUM MANGANESE MERCURY NICKEL POTASSIUM SELENIUM SILVER SODIUM STRONTIUM VANADIUM ZINC	EPA 200.7 EPA 200.7 EPA 602 EPA 206.2 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 601 EPA 200.7 EPA 239.2 EPA 200.7 EPA 200.7 EPA 200.7 EPA 245.7 EPA 200.7 EPA 200.7 EPA 270.2 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200.7 EPA 200 EPA 200.

PROGRAMS:

AW=AIR, HW=HAZARDOUS WASTE  
 AFD=SAFE DRINKING WATER, WW= WASTEWATER

STATUS:

P=PROVISIONAL  
 T=TEMP. LICENSE

*The Commonwealth of Massachusetts*



*Department of Environmental Protection  
Division of Environmental Analysis*

*Certifies*

Laboratory ID #: M-CA078

Inchcape Testing Services Environ Lab  
1961 Concourse Drive, Suite E  
San Jose, CA 95131

*for the Chemical Analysis of Potable and Non-Potable Water*

*pursuant to 310 CMR 42.00*

*Laboratory Director:* Susan Kraska Yeager

*Expiration Date:* 06/30/97

*This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.*

*This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P.*

*Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.*

  
\_\_\_\_\_  
*Director, Division of Environmental Analysis*

07/01/96  
\_\_\_\_\_  
*Issued*

COMMONWEALTH OF MASSACHUSETTS  
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List

EFFECTIVE DATE: 07/01/96

EXPIRATION DATE: 06/30/97

M-CA078 Inchcape Testing Services Environ Lab  
San Jose, CA

POTABLE WATER

- 101 Antimony
- \* 102 Arsenic
- 103 Barium
- 104 Beryllium
- 105 Cadmium
- 106 Chromium
- 107 Copper
- 108 Lead
- 109 Mercury
- 110 Nickel
- 111 Selenium
- 112 Silver
- 113 Thallium
- 114 Nitrate-N
- 116 Fluoride
- 117 Sodium
- 118 Sulfate
- 119 Cyanide
- 123 Total Alkalinity
- 125 pH
- 153 Trihalomethanes
- 154 Volatile Organic Compounds
- \* 155 1,2-Dibromoethane
- 156 1,2-Dibromo-3-chloropropane

\* Provisional Certification

COMMONWEALTH OF MASSACHUSETTS  
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List

EFFECTIVE DATE: 07/01/98

EXPIRATION DATE: 06/30/99

M-CA078 Inchcape Testing Services Environ Lab  
San Jose, CA

NON-POTABLE WATER

- 201 Aluminum
- 202 Antimony
- 203 Arsenic
- 204 Beryllium
- 205 Cadmium
- 206 Chromium
- 207 Cobalt
- 208 Copper
- 209 Iron
- 210 Lead
- 211 Manganese
- 212 Mercury
- 213 Molybdenum
- 214 Nickel
- 215 Selenium
- 216 Silver
- 218 Thallium
- 220 Vanadium
- 221 Zinc
- 222 pH
- 223 Specific Conductivity
- 224 Total Dissolved Solids
- \* 225 Total Hardness (CaCO<sub>3</sub>)
- 226 Calcium
- 227 Magnesium
- 228 Sodium
- 229 Potassium
- 230 Total Alkalinity
- 232 Fluoride
- 242 Total Cyanide
- 245 Oil and Grease
- 247 Volatile Halocarbons
- 248 Volatile Aromatics
- 249 Chlordane
- 250 Aldrin

\* Provisional Certification

COMMONWEALTH OF MASSACHUSETTS  
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List

EFFECTIVE DATE: 07/01/96

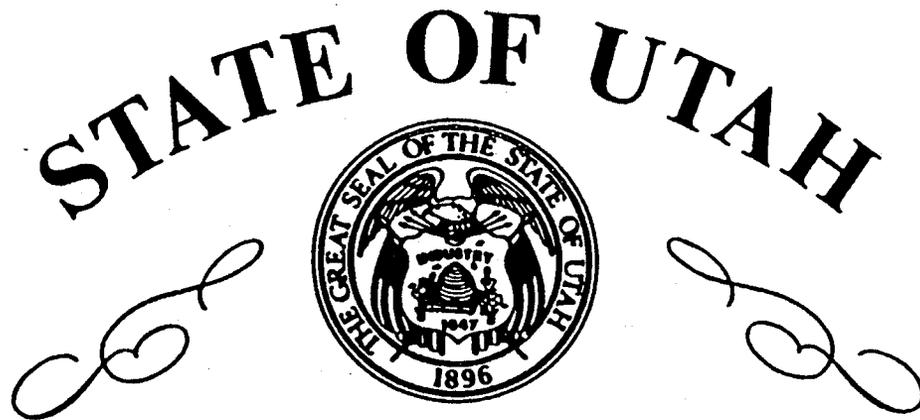
EXPIRATION DATE: 06/30/97

M-CA078 Inchcape Testing Services Environ Lab  
San Jose, CA

NON-POTABLE WATER

251 Dieldrin  
252 DDD  
253 DDE  
254 DDT  
255 Heptachlor  
256 Heptachlor Epoxide  
257 Polychlorinated Biphenyls (water)

\* Provisional Certification



# Department of Health

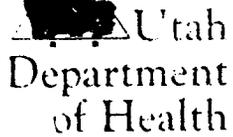
## *Inchcape Testing Services* *San Jose, California*

*having complied with the requirements of laws of the State of Utah  
and the requirements of this Department, is hereby declared a certified*

## *Environmental Testing Laboratory*

*approved to perform the analytical procedures on record at this office.  
Issued this 25th day of May, 1994      Certificate Number: E-240*

*David B. Mandenhall, Interim Director, Division of Laboratory Services*



# State of Utah

Michael G. Leavitt

Rod L. Beir

Charles D. Beckopp, Dr. PH.

Bureau of Laboratory Improvement

JUL 22 1996

SUSAN KRASKA YEAGER  
INCHCAPE TESTING SVCS - CA  
1961 CONCOURSE DR STE E  
SAN JOSE CA 95131

Customer ID: INCH2  
Account No: 4084328192

On the basis of your most recent audit results and compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Resource Conservation and Recovery Act and authorized to perform the following analytes, or groups of analytes by method:

## METALS

ALUMINUM 6010A  
ANTIMONY 6010A  
ARSENIC 6010A  
BARIUM 6010A  
\* BERYLLIUM 6010A  
CADMIUM 6010A  
CHROMIUM 6010A  
COBALT 6010A  
COPPER 6010A  
IRON 6010A  
LEAD 6010A  
MANGANESE 6010A  
MERCURY 7470A  
MERCURY 7471A  
MOLYBDENUM 6010A  
NICKEL 6010A  
SELENIUM 6010A  
SILVER 6010A  
THALLIUM 6010A  
VANADIUM 6010A  
ZINC 6010A

## MINERALS

CALCIUM 6010A  
POTASSIUM 6010A

## MISCELLANEOUS

CHLORIDE 9056  
CYANIDE TOTAL/AMENABLE 9010A  
FLUORIDE 9056  
HALOGENATED VOLATILE ORG 8021A  
LIQUID-LIQUID EXTRACTION 3510B  
LIQUID-LIQUID EXTRACTION 3520B  
NITRATE 9056  
NITRITE 9056  
ORGANOCHL PEST 8081  
SEMIVOLATILES 8270B  
SULFATE 9056  
TCLP METAL 1311  
TCLP SEMI-VOLATILE 1311  
TCLP VOLATILE 1311  
VOLATILES 8260A  
VOLATILES ORG CMPND-WATER 8021

\* Provisional Certification

This laboratory's certification is effective JUL 03 1996.

The expiration date for this laboratory's certification is MAY 31 1998. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certificate letter for the authorized method. Please call 801-584-8469.

Page Two

INCHCAPE TESTING SVCS - CA

JUL 22 1996

Resource Conservation and Recovery Act

The analytes or groups of analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. Any discrepancies must be documented and notice received by this Bureau within 15 days of receipt. The certification will be recalled in the event that your Laboratory's certification is revoked.

Respectfully,

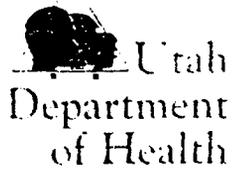


Charles Brokopp, Dr. P.H.  
Director

cc. Utah Department of Environmental Quality  
    Kevin W. Brown - Division of Drinking Water  
    Dennis Downs - Division of Solid and Hazardous Waste  
    Don A. Ostler - Division of Water Quality  
U.S. EPA Region VIII QAO

---

The expiration date for this laboratory's certification is MAY 31 1998. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certificate letter for the authorized method. Please call 801-584-8469.



# State of Utah

Michael O. Lewis

Rod L. Bell

Charles B. Hankins, Ph.D.

Director of Laboratory Operations

JUL 17 1996

SUSAN KRASKA YEAGER  
INCHCAPE TESTING SVCS - CA  
1961 CONCOURSE DR STE E  
SAN JOSE CA 95131

Customer ID: INCH2  
Account No: 4084328192

On the basis of your most recent audit results and compliance with the ELCP requirements, the laboratory listed is certified for environmental monitoring under the Clean Water Act and authorized to perform the following analytes, or groups of analytes by method:

**METALS**

- ALUMINUM 200.7
- ANTIMONY 200.7
- ARSENIC 200.7
- BARIUM 200.7
- \* BERYLLIUM 200.7
- CADMIUM 200.7
- CHROMIUM 200.7
- COBALT 200.7
- COPPER 200.7
- IRON 200.7
- LEAD 200.7
- MANGANESE 200.7
- MERCURY 245.1
- MOLYBDENUM 200.7
- NICKEL 200.7
- SELENIUM 200.7
- SILVER 200.7
- THALLIUM 200.7
- VANADIUM 200.7

ZINC 200.7

**MINERALS**

- CALCIUM 200.7
- POTASSIUM 200.7
- SODIUM 200.7

**RESIDUE**

- RESIDUE FILTERABLE TDS 160.1
- RESIDUE TOTAL 160.3

**ORGANIC**

- PURGEABLE AROMATIC 602
- PURGEABLE HALOCARBONS 601

**INORGANIC**

- CYANIDE 335.2
- CYANIDE AMENABLE 335.1
- TURBIDITY 180.1

\* Provisional Certification

The effective date for this certification is JUL 03 1996.

The expiration date for this laboratory's certification is MAY 31 1998. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certificate letter for the authorized method. Please call 801-584-8469.

Page Two  
INCHCAPE TESTING SVCS - CA  
JUL 17 1996  
Clean Water Act

The analytes or groups of analytes by method which a laboratory is authorized to perform at any given time will be those indicated in the most recent certificate letter. The most recent certification letter supersedes all previous certification or authorization letters. Any discrepancies must be documented and notice received by this Bureau within 15 days of receipt. The certification will be recalled in the event your Laboratory's certification is revoked.

Respectfully,



Charles Brokopp, Dr. P.H.  
Director

cc. Utah Department of Environmental Quality  
Kevin W. Brown - Division of Drinking Water  
Dennis Downs - Division of Solid and Hazardous Waste  
Don A. Ostler - Division of Water Quality  
U.S. EPA Region VIII QAO

---

The expiration date for this laboratory's certification is MAY 31 1998. The Utah Environmental Laboratory Certification Program (ELCP) encourages clients and data users to verify the most current certificate letter for the authorized method. Please call 801-584-8469.

# SCOPE OF ACCREDITATION

Inchcape Testing Services - Anametrix  
San Jose, California

is accredited by the State of Washington Department of Ecology to perform analyses for the parameters listed below using the analytical methods indicated. This Scope of Accreditation applies to non-potable water analyses only. Accreditation for all parameters is final unless indicated otherwise in a note. EPA refers to the U.S. Environmental Protection Agency. "SM" refers to APHA *Standard Methods for the Examination of Water and Wastewater*, 18th Edition.

<u>PARAMETER</u>	<u>METHOD</u>	<u>NOTES</u>
Alkalinity	EPA 310.1	
Ammonia	EPA 350.3	
Anionic Surfactants	EPA 425.1	1
Bromide	EPA 300.0 A	1
Calcium	EPA 200.7/6010	
Chloride	EPA 300.0 A	
Cyanide Total	EPA 9010	
Cyanides Amenable to Chlorination	EPA 335.1+335.2(8.7)	
Fluoride	EPA 340.2	
Hardness Total	SM 2340 B	
Magnesium	EPA 200.7/6010	2
Nitrate	EPA 300.0 A	
Nitrite	EPA 300.0 A	1
pH	EPA 9040	
Potassium	EPA 200.7/6010	
Sodium	EPA 200.7/6010	
Solids Total Dissolved	EPA 160.1	
Specific Conductance	EPA 120.1	2
Sulfate	EPA 300.0 A	
Sulfide	EPA 376.1	1
Sulfide	EPA 9030	
Turbidity	EPA 180.1	

<u>PARAMETER</u>	<u>METHOD</u>	<u>NOTES</u>
Aluminum	EPA 200.7/6010	
Antimony	EPA 200.7/6010	
Arsenic	EPA 206.2/7060	
Arsenic	EPA 200.7/6010	
Barium	EPA 200.7/6010	1
Beryllium	EPA 200.7/6010	
Cadmium	EPA 213.2/7131	
Cadmium	EPA 200.7/6010	
Chromium	EPA 218.2/7191	
Chromium	EPA 200.7/6010	
Cobalt	EPA 200.7/6010	
Copper	EPA 220.2/7211	
Copper	EPA 200.7/6010	
Iron	EPA 200.7/6010	
Lead	EPA 239.2/7421	
Lead	EPA 200.7/6010	
Manganese	EPA 200.7/6010	
Mercury	EPA 245.1/7470	
Molybdenum	EPA 200.7/6010	
Nickel	EPA 200.7/6010	
Selenium	EPA 270.2/7740	
Selenium	EPA 200.7/6010	
Silver	EPA 272.1/7760	
Silver	EPA 200.7/6010	
Thallium	EPA 279.2/7841	
Thallium	EPA 200.7/6010	
Tin	EPA 200.7	1
Tin	EPA 6010MOD	1,3
Titanium	EPA 200.7	
Vanadium	EPA 200.7/6010	
Zinc	EPA 200.7/6010	
Organochlorine Pesticides	EPA 8081	
Polychlorinated Biphenyls	EPA 8081	
Phenols	EPA 8040	
Purgeable Aromatics	EPA 602	
Purgeable Halocarbons	EPA 601	
Purgeable Halocarbons	EPA 8021	

<u>PARAMETER</u>	<u>METHOD</u>	<u>NOTES</u>
BNA Extr (Semivolatile) Organics	EPA 8270	
Purgeable (Volatile) Organics	EPA 8260	

- NOTES: (1) Interim pending ability of Department of Ecology to identify acceptable performance evaluation sample (WAC 173-50-100).  
(2) Provisional pending receipt of fully acceptable performance evaluation sample analysis results (WAC 173-50-110).  
(3) Method modified to ensure digestion and quantification of metal which is not include in method.

AUTHENTICATION: Cliff J. Kirchner  
Cliff J. Kirchner, Ph.D.  
Quality Assurance Officer

May 26, 1997  
Expiration Date

## APPENDIX I

**Inchcape Testing Services  
Intercorporate Work**

From Lab Name: \_\_\_\_\_  
 From Lab Project Manager: \_\_\_\_\_  
 Date Project To Start: \_\_\_\_\_  
 Duration: \_\_\_\_\_

To Lab Project Manager: \_\_\_\_\_  
 Project Name: \_\_\_\_\_  
 Purchase Order/Work Order: \_\_\_\_\_

Scope of Work (Attach compound list and detection limits):

# Samples	Matrix	Analysis	Method	Client Price	Sub- Lab Price

Bill  Client  Lab

Send Results to Lab:  Yes  No  
 ATTN: \_\_\_\_\_

Report Due Date: \_\_\_\_\_  
 Special Comments: \_\_\_\_\_

Send Results to Client:  Yes  No  
 Client Name: \_\_\_\_\_  
 Client Address: \_\_\_\_\_

#Copies of Report Required: \_\_\_\_\_

Phone # \_\_\_\_\_  
 Fax # \_\_\_\_\_

Expected Level of analysis:  High  Moderate  Low  Unknown

**QC Reporting Level: (Choose one)**

- Results, Surrogate Recovery  Matrix Spikes. Billable:  Yes  No
- Blank Spikes. Billable:  Yes  No  MSD Billable:  Yes  No
- Results, Surrogates, Blank Spike Results (MSB or LCS), copy of COC, Narrative
- CLP Forms 1-4, log-in anomaly sheet, copy of COC, Narrative
- Full CLP Level 4 (all of above, plus raw data- chromatograms, etc)
- Other, Please specify \_\_\_\_\_

**Reporting:**

- Report Reanalysis. Billable:  Yes  No
- Special Report Limits/Compounds-Attach List **or**  Lab Std Report Limits
- % Solids needed **or**  %Lipids needed
- Report Soils/Tissues on an "as received" basis **or**  Report Soils on "dry weight" basis
- Electronic Deliverable. Type \_\_\_\_\_

**APPENDIX K**

**QAPP**

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# QUALITY ASSURANCE PROJECT PLAN (QAPP)

## 1. PURPOSE OF THE QUALITY ASSURANCE PROJECT PLAN

This Quality Assurance Project Plan (QAPP) has been prepared to document the quality assurance protocols for execution of this Pilot Scale, Treatability Study. The purpose of this QAPP is to define the field and laboratory data requirements for the Treatability Study as specified in the Field Sampling Plan and to ensure that the data are of sufficient quality to support the end use of the data. The QAPP defines the policy, organization, functional activities, and quality assurance (QA) and quality control (QC) protocols that will be used to meet the data quality objectives (DQOs) of this investigation. Description of all of the DQOs and procedures associated with the field programs, including sample collection, sample custody, laboratory analysis, and QAPP for this project are described in this document. Adherence to the procedures described in this QAPP should generate data that are scientifically sound, valid, defensible, and of known, acceptable, and documented quality.

## 2. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT

### 2.1 Data Quality Objectives

The overall quality assurance objective for this investigation is to develop and implement sampling, sample handling, and analytical procedures that will provide data that can be used to fulfill the data quality objectives (DQOs) as stated in the Work Plan. DQOs are qualitative and quantitative statements developed by data users to specify the quality of data from field and laboratory data collection activities that is needed to support specific decisions or regulatory actions. The DQOs describe which data are needed, why the data are needed, and how the data will be used to meet the needs of the project. DQOs also establish numeric limits for the data to allow the data user (or reviewers) to determine whether the data collected are of sufficient quality for their intended use.

DQO development as described in USEPA guidance is based on:

- Identifying project objectives
- Specifying the data necessary to meet project objectives
- Describing the methods that will yield data of acceptable quality and quantity to support the required decisions

The project objectives are stated in the Introduction and data specifications are described in the Field Sampling Plan. Analytical and testing methods are described in this QAPP. Table 1-1 and 1-2 lists the DQOs for this sampling program.

Table 1-1. DQO's for Performance Parameters

Parameter	MDL μg/L	Accuracy %E @ RO	Precision %RSD
TCE	0.02	30	<15
1,1DCE	0.05	30	<30
t1,2 DCE	0.03	30	<15
c1,2 DCE	0.06	30	<15
VC	0.04	35	<30
Benzene	0.03	30	<15
Toluene	0.08	30	<30
Ethylbenzene	0.03	30	<30
p-Xylene	0.06	30	<15
m-Xylenes	0.06	30	<15
pH	pH range of 5-12	+/- 0.2 pH units	<0.2
Fe	.	+/- 20%	<30

Table 1-2. DQO's for Treatment Parameters

Parameter	MDL µg/l	Accuracy % E @ RO	Precision % RSD
TCE	5	30	<30
1,1 DCE	7	30	<30
t1,2 DCE	20	30	<30
c1,2 DCE	20	30	<30
VC	5	30	<30
Benzene	2	30	<30
Toluene	5	30	<30
Ethylbenzene	5	30	<30
m + p-Xylenes	5	30	<30
o-Xylene	5	30	<30
ethene, methane	20 each	30	<30
DO	0.2 mg/l	30 @ 2 mg/l	<50 @ 2 mg/l
alalkinity	1 mg/l	30	<30
other inorganics	0.1 mg/l	20	<30
BOD	5 mg/l	30	<30
Fe	0.05 mg/l	20	<30
EC	0.5 µs	+/- 50 µs	<20

## **2.2 Analytical Quality Control Levels**

Five levels of analytical quality control are identified by CERCLA and are described in *Data Quality Objectives for Remedial Response Activities Development Process* (USEPA, 1987). These levels are based on the type of site under investigation, the required precision and accuracy, the end use of the analytical data, and the level of documentation. Three levels of analytical data will be collected during this investigation.

Level I - Level I analysis provides data for onsite, real-time total vapour measurements, evaluation of existing conditions, optimal sample locations, extent of contamination, and health and safety evaluations. Data generated from Level I are qualitative or semi-qualitative data obtained by use of approved field equipment such as total organic vapour analysers, dissolved oxygen meters, pH, conductivity and temperature meters. The most available form of documentation for this support level is the field operator log book. Sample identification, location, instrument reading, calibration and blank information is usually contained in the field log book.

Level II - Level II analytical support is designed to provide real-time qualitative data for ongoing field activities, real time screening, extent of contamination, on site remedial activities or when initial data will provide the basis for seeking laboratory analytical support. Field analysis involves the use of portable or transportable instruments which are based at or near a sampling site. Typically, an In Situ Permeable Flow Sensor or a GeoFlow meter operated in the field provide the bulk of the analytical support at this level. Documentation of this type of data would consist of the output of an integrator or strip chart recorder for all samples, standards, and blanks analysed and the field operator log book.

Level III - Level III data are quantitative, have known precision and accuracy, and are produced under controlled conditions using laboratory-grade instrumentation. Level III protocols all have built-in QA/QC, including calibration runs, surrogate standards, etc. External QA is employed in the form of trip blanks, replicate and duplicate samples, and blind spikes submitted with the samples. Generally the analyses performed are designed to provide confirmed identification and quantification of organic and inorganic compounds in water, sediment, and soil samples. This data is used for site characterization, environmental monitoring, confirmation of field data and to support engineering studies (i.e design, modelling, and pilot/bench studies).

## **2.3 Data Quality Definition and Measurement**

The effectiveness of a QA program is measured by the quality of data generated in the field and by the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness, and comparability. These terms are described in the following sections.

### 2.3.1 Accuracy

Accuracy is the degree of agreement of a measurement or an average of measurements with an accepted reference or “true” value, and is a measure of bias in the system. The accuracy of a measurement system is impacted by errors introduced through the sampling process, field contamination, preservation, handling, sample preparation and analytical techniques.

Accuracy is evaluated by the following equation:

$$\%R = 100\% * \left[ \frac{|C_{sm} C_m|}{C_{sm}} \right]$$

where:

- $\%R$  = percent recovery
- $C_m$  = measured concentration of standard reference material
- $C_{sm}$  = actual concentration of standard reference material

For performance samples, accuracy will be assessed and controlled by the results of the following QC samples, which contain known concentrations of specific analytes (spiked):

- Matrix spike (MS) and matrix spike duplicates (MSD)
- Laboratory control samples (LCS) and LCS duplicates (LCSD)
- Surrogate spikes

Treatment samples be assessed for accuracy using LCS and surrogate spikes. As these are analysed, spike recoveries will be calculated and compared to acceptance limits agreed upon by the University’s Organic Geochemistry Lab and the QC officer. For the Navy certified lab, QA/QC reports with spike recoveries will be provided to the QC officer. For the University’s Organic Geochemistry lab, MS/MSD and surrogate samples will not be analysed for accuracy. Acceptance limits are based on previously established laboratory performance or specified by the analytical methods. The control limits reflect the minimum and maximum recoveries expected for individual measurements for an in-control system. Recoveries outside the established limits indicate error in addition to normal measurement error and the possible need for corrective action. Corrective action may include re-calibrating the instrument, reanalysing the QC samples, reanalysing the sample batch, re-preparation of the sample batch, or flagging the data (if problems can not be resolved). For contaminated samples, matrix spike recoveries may be dependent upon sample homogeneity, matrix interference, and dilution requirements.

### 2.3.2 Precision

Precision is the reproducibility of measurements under a given set of conditions. For large data sets, precision is expressed as the variability of a group of measurements compared to their average value (i.e. standard deviation). For duplicate measurements, precision is expressed as the relative percent difference (RPD) of a data pair and is calculated using

$$RPD = \frac{(C_1 C_2) * 100\%}{(C_1 + C_2) / 2}$$

the following equation: differ

- where:
- RPD = relative percent ence
  - C<sub>1</sub> = larger of the two observed values
  - C<sub>2</sub> = smaller of the two observed values

For performance samples, precision will be assessed by calculating the RPD of the MS/MSD sample pairs and the blind field replicates and field duplicate sample pairs and comparing the results to laboratory established RPD control limits, which are listed in Table 1-1 for performance data. The RPD for the treatment samples will be calculated using blind field replicates and field duplicate sample pairs. These values will be compared to the established control limits listed in Table 1-2. The University of Waterloo's Organic Geochemistry Lab may use LCS for precision analysis as well. Precision of blind field replicate samples is dependent upon sample homogeneity.

The analyst, group leader, or technical advisor is responsible for investigating data outside the QC limits. Corrective action may include re-calibrating the instrument, reanalysing QC samples, re-analysing samples or flagging data.

### 2.3.3 Representativeness

Representativeness is a qualitative expression of the degree to which sample data accurately and precisely represent a characteristic of a population, a sampling point, or an environmental condition. Representativeness is maximized by ensuring that, for a given project, the number and location of sampling points and the sample collection and analysis techniques are appropriate for the specific investigation, and that the sampling and analysis program will provide information that reflects "true" site conditions. Results for blind duplicate sample analysis are also used to evaluate representativeness.

### 2.3.4 Comparability

Comparability is a qualitative parameter that expresses the confidence that one data set may be compared to another. Comparability of data is achieved through the use of

standardized methods for sample collection and analysis, and the use of standardized units of measure.

### 2.3.5 Completeness

Completeness is defined as the percentage of valid data relative to the total number of analytes and is evaluated using precision, accuracy, and holding time criteria. Completeness is calculated by the number of samples with acceptable data divided by the total number of samples planned to be collected, and multiplied by 100.

Project completeness is determined at the conclusion of the data validation. While 100 percent completeness is the goal, the project will be considered to have met its objectives with 90 percent completeness. If completeness is less than 90 percent, UW will provide documentation explaining why this objective was not met and the impact, if any, of a lower percentage on the project.

## **3. SAMPLING PROCEDURES**

All of the sampling locations and procedures to be used for environmental sample collection are presented in the Sampling and Analysis Plan, section 6. Section 6.0 describes in detail the procedures that will be followed during sampling (for both treatment and performance) to ensure that the data are representative of environmental conditions. The remainder of this section describes the sampling procedures that will be used to collect QC samples in the field. The types of sample containers, preservation required for each matrix, holding times, and volume are listed in table 6-3 for the respective sampling programs.

### **3.1 Equipment Blanks**

Equipment blanks are used to identify sources of contamination from 1) non-disposable sampling equipment, 2) previously-collected samples, or 3) conditions during sampling. Equipment blanks will consist of distilled or deionized water poured over or through any equipment which comes into contact with field samples. Equipment blanks will be collected at a rate of one per day when non-dedicated or non-disposable equipment is used for sampling. Equipment blanks will be collected for each analytical parameter for which the associated environmental sample was collected. All samples will be labelled, handled and shipped following the procedures outlined in Section 4.0 of this QAPP.

### **3.2 Field Blanks**

Field blanks will be collected to assess potential contamination of samples during sample collection by dust or other sources at the site. During field operations, one or more sample containers will be filled with organic-free, distilled, deionized water. Field blanks will be collected in the vicinity of the gate. Field blanks will be preserved as specified in the FSP.

### **3.3 Blind Field Replicates**

One blind field replicate sample will be submitted during each sampling event or for each day if a sampling event goes beyond one day. A blind field replicate sample is a single grab sample (250 ml) that is split into three samples during collection. One of the samples will be labelled with the correct sample identification and the other 2 samples will be labelled with a false sample identification. The location of the falsely identified samples will be recorded in the field log book. Both samples will be sent to the same laboratory for analysis. The samples will be labelled, handled, and shipped following the procedures outlined in Section 4.0 of this QAPP.

### **3.4 Matrix Spike and Matrix Spike Duplicate Samples**

Samples for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be collected for five percent of the total number of performance samples and scheduled analytical method. There will be no MS and MSD sampled collected for any treatment sampling. The same procedures used to collect blind replicate samples during sampling will be used to collect samples for MS/MSD analysis.

### **3.5 Trip Blank**

Trip blanks will be prepared by the laboratory prior to sampling and will consist of two 40 mel VOA bottles filled with preserved reagent grade water. The bottles will be filled so that there is no head space and will be capped with a Teflon septum. Trip blanks will accompany all samples scheduled for VOC analysis.

### **3.6 Field Duplicates**

All CVOC samples (treatment and performance) will be routinely collected as duplicates. Normally, only one of the duplicates will be analysed, but from each round of sampling, 2 sets of duplicates will be analysed to assess variability associated with collected discrete field samples.

## **4. SAMPLE CUSTODY, HANDLING, AND SHIPPING PROCEDURES**

To ensure that samples are identified correctly and remain representative of the environment, the documentation and sample custody procedures specified in this section will be followed during sample collection and analysis. Standard sample documentation and custody procedures, as outlined below, will be used during each sampling program to maintain and document sample integrity during collection, transportation, storage, and analysis. The Field Team Leader, to be designated at the time of the investigation, will be responsible for ensuring proper documentation and custody procedures are initiated at the time of sample collection, and that individual samples can be tracked from the time of sample collection until the samples are relinquished to the laboratory. The laboratory will be responsible for maintaining sample custody and documentation from the time the

samples are relinquished to the lab until final sample disposition.

#### **4.1 Chain of Custody**

Chain of custody (COC) procedures provide an accurate written record of the possession of each sample from the time of collection in the field through laboratory analysis. A procedure will be defined to maintain and document the COC for all samples, once the facilities at the field site are known. A sample is considered in custody if one of the following applies:

- It is in an authorized person's immediate possession
- It is in view of an authorized person after being in physical possession
- It is in a secure area after having been in physical possession of an authorized person
- It is in a designated secure area, restricted to authorized personnel only

#### **4.2 Field Procedures**

The sample custody and documentation procedures will be initiated at the time of sample collection. Sample collection details will be documented on the groundwater sampling forms. Samples will be labeled and the appropriate information will be recorded on the COC form using indelible ink. Any errors will be corrected by drawing a single line through the incorrect entry, entering the correct information, and then initialling and dating the change.

#### **4.3 Sample Labels and Identification**

##### 4.3.1 Sample Labels

Sample labels will be completed and attached to sample containers at the time of sample collection. The following information will be included on the sample label:

- Project name/location
- Sample location
- Field sample identification (see below)
- Date of sample collection
- Type of analysis to be performed
- Preservative (if applicable)

##### 4.3.2 Sample Identification

Each sample will be identified with an alphanumeric code. These abbreviations will be as

follows:

<u>Sample Code</u>	<u>Definition</u>
R	Row
#	Row number (see Figure6-1)
P	Performance well
T	Treatment well
A, B or C	Column designation
1,2 or 3	Depth (Treatment wells only)

Treatment wells consist of a nest of three multilevel, 1/8" piezometers. The shallowest depth is represented as a 1, the next depth down from ground surface as a 2 and the deepest depth as a 3. Performance wells are fully screened hence there is no depth distinction. A typical sample identification for a treatment well and a performance well for row 1, well A is:

R1TA-1

R1PA

For the control gate, all the wells are performance monitoring wells and will be designated with a prefix C. The only rows that these wells are located in are 1,5 and 8. A typical sample identification for a performance well in the control gate for row 1 is:

CR1

#### **4.4 Chain of Custody Record**

Properly completed COC forms will ensure that sample custody is documented, appropriate sample fractions have been collected, and scheduled analyses are properly assigned. Unused portions of the COC form will be crossed out and initialled. A completed COC record will be included with each sample cooler. The sampler will retain a copy of the COC. When shipping the sample cooler to either laboratory by a commercial carrier, the COC will be signed, placed in a plastic bag, and taped to the inside of the shipping container used for sample transport. Signed air bills will serve as evidence of chain of custody transfer between the field sampler and courier, and courier and laboratory. The sampler will retain and file copies of the COC record and the air bill after the samples are shipped. The samples are relinquished to the laboratory upon arrival and the laboratory personnel then will complete the COC.

#### **4.5 Laboratory Custody Procedure**

Upon receipt in the laboratory, the integrity of the shipping container will be checked. The sample containers will be checked for breakage, leakage, damage, and the contents of the shipping container will be verified against the COC. Container integrity, cooler temperature and sample preservation will be documented on the sample control worksheet.

A permanent logbook will be maintained in the sample control area to document the following:

- Date of sample receipt
- Lab sample submission
- Number of samples
- Source of samples

All insufficiencies and/or discrepancies will be immediately reported to the laboratory project manager. The laboratory project manager will either resolve the problem internally or contact the project manager at UW.

Once the samples have been recorded in the permanent logbook, the samples will be transferred to the appropriate refrigerators in the lab. The sample refrigerators will be kept at  $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$ .

#### **4.6 Sample Handling and Shipping**

After each water sample is collected, it will be placed in a cooler containing blue ice, and the cooler will be shipped by overnight courier to the University of Waterloo's Inorganic Geochemistry laboratory or to a commercial laboratory. The samples will be placed upright in the cooler, and secured with inert cushioning material to prevent breakage. A completed COC form will accompany all samples. Complete packaging and shipping procedures are as follows:

- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- Blue ice will be placed around, among, and on top of the sample bottles. Enough blue ice will be used so that the samples will be maintained at  $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$ .
- To prevent the sample containers from sliding around the cooler, the cooler will be filled with inert cushioning material such as shipping peanuts, additional bubble pack, or cardboard dividers.
- The completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid.
- The lid will be secured with strapping tape by wrapping it completely around the

cooler.

- The completed shipping label will be attached to the top of the cooler and “This side up” and “Fragile” labels will be placed on all sides of the cooler.

#### **4.7 Sample Disposal**

Thirty days after a laboratory report has been generated (from both the UW’s Organic Geochemistry Lab and the external lab) and submitted to UW, the samples are transferred to the sample disposal area. Samples will be disposed according to each laboratory’s SOP.

### **5. CALIBRATION PROCEDURES**

This section discusses general requirements for field equipment and laboratory instrument calibration and standards preparation. Instrument calibration is necessary for accurate sample qualification, and establishing the dynamic range of the instrument. The following paragraphs outline the calibration procedures for the field equipment and laboratory instrumentation.

#### **5.1 Field Equipment**

The field equipment to be used during the groundwater sampling program include a water-level sounder, a pH meter, dissolved oxygen, temperature and EC meter. The meters will be calibrated according to the procedures outlined below.

##### 5.1.1 Water-Level Sounder

Electric water-level sounders will be checked before the beginning of field activities by comparing the scale on the water-level tape against an engineering measurement tape.

##### 5.1.2 pH Meter:

Criteria for calibration of the pH meter is specific to instrument manufacturer. This meter will be calibrated at the start and end of each day using 3 point standard calibration curves.

##### 5.1.3 EC:

Criteria for calibration of the EC meter is specific to instrument manufacturer. This meter will be calibrated at the start and end of each day using electrical conductivity standards.

##### 5.1.4 Dissolved Oxygen

Criteria for calibration of the dissolved oxygen meter (DO) is specific to instrument manufacturer. At minimum calibration of the DO meter will be calibrated at the start and

end of each day and at any time in which the operator observes instrument drift.

#### 5.1.5 Temperature:

There is specific calibration procedure for the temperature meter.

### **5.2 Laboratory Instruments**

An external laboratory, Inchoape Testing Services (ITS) of San Jose, CA as well as the UW's Organic Geochemistry Lab will perform analytical services for all Level III data. The following paragraphs describe procedures for standard preparation and instrument calibration from UW's Organic Geochemistry Lab. The procedures used by ITS are described in their SOP which is presented in Appendix G.

#### 5.2.1 Standard/Reagent Preparation

Data accuracy is dependent upon the accuracy of the standards used for instrument calibration. To ensure the highest quality standard, primary reference standards used by the UW laboratories, will be obtained from the National Institute of Standards and Technology (NIST) or other reliable commercial sources. When standards are received at the laboratory, the date received, supplier, lot number, purity, concentration, and expiration date are recorded in a standards log book. Vendor certification for the standards are retained in the lab's files.

Standards are obtained either in their pure form, or in stock or working standard solutions. Dilutions are made from vendor standards. All standards are given a standard identification number and the following information is recorded in the standards log book; source of the standard, the initial concentration of the standard, the final concentration of the standard, the volume of the standard that was diluted, the volume of the final solution, the solvent and the source and lot number of the solvent used for standard preparation, and the preparer's initials. All standards are validated prior to use.

Validation procedures for standards include a check for purity and verification of the standard's concentration by comparing its response to a standard of the same analyte prepared at a different time or obtained from a different source. Reagents also are analysed for purity; for example, every lot of dichloromethane (used for organic extraction) is analysed for contaminants prior to use in the laboratory. Standards are checked routinely for signs of deterioration (e.g discolouration, formation of precipitates, and changes in concentration) and are discarded if deterioration is suspected or the expiration date has passed. Expiration dates are based on vendor recommendation, the analytical method, or internal research.

Calibration of instruments to be used in the University's Organic Geochemistry Laboratory will be conducted in one of two ways, depending on the stability of the instrumentation. The first procedure will involve preparation of calibration stock solutions

from either analytical grade salts, solutions or neat organic liquids. These will be diluted to obtain at least three calibration standards which can be used to construct a calibration line. In the case of volatile organics, these solutions will be prepared daily. The standards will be analyzed to verify that the calibration line is linear, and they will be included in the analytical batches (1 standard for every 10 to 15 samples) to verify that instrument drift is not occurring systematically. The data from all the standards will be used to construct the final calibration lines for each analytical batch, and the confidence intervals for that line will be calculated using the method of Taylor (1987), with equations from Snedecor and CVOChran (1989) and Anderson (1987) (Appendix J). Concentrations and uncertainties will be determined from the calibration line and confidence intervals. If uncertainties exceed DQOs, then corrective action will be taken and samples re-run, if possible. This could involve re-calibration and re-running samples.

The second procedure will apply to instrumentation shown to be stable over time. Complete calibration curves will only be determined biweekly to monthly. A standard will be inserted in the analytical batches (1 standard for every 20 to 30 samples) to verify that instrument drift is not occurring systematically. Final calibration curves for each batch will be constructed from the most recent complete calibration curve and the subsequent batch standards. There will be at least three standards and a blank per range. Concentrations and uncertainties will be determined as described above.

#### 5.2.2 Calibration of Organic Methods

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the sensitivity necessary to meet established reporting limits. Each instrument will be calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method.

Analytical instruments will be calibrated using standards in accordance with the specified analytical methods and manufacturer's procedures. At a minimum, written calibration procedures include the equipment to be calibrated, the reference standards used for calibration, the calibration techniques, actions, acceptable performance tolerances, frequency of calibration, and calibration documentation format. Records of standard preparation and instrument calibration will be maintained. Instrument calibration will include daily checks using standards prepared independently of the calibration standards and instrument response will be evaluated against established criteria. The analysis logbook, maintained for each analytical instrument, will include at a minimum: the date and time of calibration, the initials of the person performing the calibration, the calibrator reference number and concentration. The QAPP for the the external lab is included as Appendix I. The University's Organic Geochemistry lab does not have an official QAPP but does follow specific calibration procedures.

## **6. ANALYTICAL PROCEDURES AND DETECTION LIMITS**

All samples collected for Level III data will be analysed by either the University of

Waterloo's Organic Geochemistry lab (Treatment samples) or an external analytical laboratory (Performance samples). Some volatile organic analyses of treatment samples may also be conducted by the external laboratory, if the desired MDL is available in that laboratory when it is not available at Waterloo. The samples will be analysed using the methods listed in Table 6-1 (of this QAPP) for the treatment and performance samples. The units of measure and typical limit of quantification for each analyte are also listed in Table 6-1 for both the treatment and performance samples. These limits of quantification can be met in the absence of matrix interferences or high contaminant concentrations, and are at least as stringent as the reporting limits specified for the individual analytical methods. The laboratory-specific analytical methods are detailed for both the external laboratory (Performance samples) and the University's Organic Geochemistry lab (Treatment samples) and in Appendix G and H respectively.

Table 6-1: Analytical methods for specific analytes

Analytes	Analytical Method	Units of Measure
CVOC's OGL Navy Certified Lab	Headspace EPA SW-8260	µg/L
ethene, ethane and methane	GC-FID	µg/L
cations	ICP	mg/L
anions	Dionex Exchange Column	mg/L
DO	Probe or Chemetric	mg/L
BOD	APHA, -----	mg/L
EC	probe	uS
pH	probe	none
alkalinity	titration	mg/L

Note: LOQs will be established (typically <2x MDL) for each analyte.

## 7. DATA REDUCTION, VALIDATION, AND REPORTING

Procedures must be used to ensure that all laboratory data generated and processed are scientifically valid, defensible, and comparable. The following sections describe the data reduction, validation, reporting procedures, and reporting responsibilities for chemical analyses for this demonstration project. Data from the external lab will be visually inspected and if deemed necessary, it will be entered into the FORTRAN program where it will be reduced and validated. All data from the treatment sampling will be sent to the University of Waterloo where it will be reduced and validated according to methods described in section 7.2.1 and 7.2.2 (following).

### 7.1 Field Measurements

Raw data from field measurements and sample collection activities will be documented in the field log book and on the appropriate forms, as described in the QAPP, section. Field analyses not requiring calibration (water levels) require no further data reduction. Where calibration is required, the calibration procedure will be followed and results noted in

specially prepared field notes. Typically, these will lay out the required calibration procedure with results to be entered. There will be clear limits for calibration values and corrective action suggested for typical out of range calibration incidences. All field measurements and data collection during sampling will be presented in a final report. All field data generated during this investigation will be evaluated under the direction of the project manager before it is incorporated in the report.

## **7.2 Laboratory Measurements**

### 7.2.1 Data Reduction Calculations

Laboratory data from the University of Waterloo's Organic Geochemistry lab will be transferred from the instrument reporting devices (integrators etc.) to a FORTRAN program which will analyse the calibration curves (see section 5.2.1) and calculate concentrations and uncertainties (Devlin, 1996). These reports will be supplied on diskette to the project manager who will transfer the data to a project database from which tables and charts will be generated. Data collection from automated equipment will be transferred to the project database electronically. Data from the external lab will be visually inspected and if deemed necessary, it will be entered into the FORTRAN program where it will be reduced.

### 7.2.2 Data Validation

The majority of treatment data generated from this project will be in the form of breakthrough curves and time series. For the University's purposes, the performance data will also be a similar form. The validation of data in this form is facilitated by visually inspecting charts to identify outlying points, then double checking the values (rechecking chromatograms and calculations, and re-analysis when possible). The results of the QA/QC sampling program will also be considered in assessing the validity of suspect data points. The Laboratory Project Manager and Laboratory QA Officer also are responsible for assessing the data quality and qualifying any data that may be unreliable. The University's lab and the external lab will prepare and retain full analytical and QC documentation.

The laboratory review of the data includes assessing compliance with the control limits in QAPP. Accuracy and precision are the primary data parameters that can be used to calculate control limits. Data to evaluate accuracy are obtained primarily from separately prepared laboratory QC samples or from spiked field samples (performance data only). Data used to evaluate precision are QC sample analyses or the replicate analysis of field samples. The calculations that are used to evaluate precision and accuracy are defined in the laboratory's SOP and/or QA/QC manual. Precision and accuracy quality control limits are generated from the statistical analysis of QC sample results. As stated in section 7.2.1, the model proposed in Devlin, 1996, will be used to determine the data quality which will be compared to the data quality limits stated in Table 1-1 and 1-2.

### 7.2.3 Data Reporting

The analytical data will be reported in a format organized to facilitate data evaluation. All of the data used by the University including QC data, performance and treatment data will be reported in the form of break through curves and time series. The following information will be included in each data package from both the University's Organic Geochemistry lab and the external lab:

- A list of diluted samples including their dilution factors
- A report for each completed environmental and QC sample analysis (equipment blanks, MS/MSD samples (performance data only), laboratory control samples, surrogate spike samples, and method blanks) that includes the following information: the field sample ID number (if applicable), the laboratory ID number, the date the sample was collected, the date the sample was received by the laboratory, the date and method of sample extraction (if applicable), the date and method of sample analysis, tabulated results for each sample, surrogate spike recoveries (if applicable), internal standard recoveries (if applicable), associated method blank results, and the detection limit for each analyte. The initial concentration of the surrogate spikes, matrix spikes, and laboratory control sample spikes, as well as the percent recovery and acceptance limits of each spiked analyte also should be reported. The samples analysed in association with each QC sample should also be identified on the report. All questionable data should be flagged and brought to the attention of the UW's project manager.
- A corrective action summary that identifies all analytical irregularities (i.e missed holding times, poor analytical recoveries), and the corrective action taken by the laboratory for the affected samples.

## **8. INTERNAL QUALITY CONTROL**

### **8.1 Field Program**

Internal quality control evaluates whether a method is performing within acceptable limits of precision and accuracy. On the sampling level, quality control samples used to assess field sampling techniques and environmental conditions during sample collection and transportation include blind field replicates, trip blanks, equipment blanks, field blanks and field duplicates.

Blind field replicates and field duplicate samples will be used to assess variability in the sample matrix and to assess sampling precision. The sampling procedures will be evaluated by comparing the analytical results of blind field replicate samples and of the field duplicate sample pairs. If the reported values for the sample pairs are similar, the samples are considered to be representative of the environment. A large difference (greater than 40 percent), between the reported values for the sample pair indicates that there may have been a problem during sampling or analysis. Blind field replicates and field duplicate analyses will also be used to evaluate precision by calculating the RPD between

the blind field replicate sample and its associated environmental sample. The RPD will be compared to the MS/MSD limits for precision for performance data only. Relative percent difference values within the QC guidelines indicate that good sampling and analytical procedures were followed. Relative percent difference values outside the QC limits indicate that sample may be heterogeneous, or that there may have been a problem during sampling and/or analysis. Section 3.3 outlines the procedures for collecting blind duplicate samples.

Trip blanks will be used to evaluate representativeness by assessing whether CVOCs were introduced into samples during handling, shipping, or storage at the laboratory. Trip blanks prepared by the laboratory (see Section 3.5) will be included with each sample shipment that contains groundwater samples for CVOC analysis.

Equipment blanks will be used to assess the equipment decontamination procedures and evaluate whether the samples are representative of the environment. The results of each equipment-blank analysis will be reviewed for the presence of target analytes. If target analytes are found, the data from the associated environmental samples will be evaluated to determine if they are representative of environmental conditions or the result of incomplete equipment decontamination. Equipment blank samples will be collected as outlined in Section 3.1.

## **8.2 Laboratory Analysis**

The general objectives of a laboratory QC program are to:

- Ensure that all procedures are documented, including any changes in administrative and/or technical procedures
- Ensure that all analytical procedures are validated and conducted according to method guidelines
- Monitor the performance of the laboratory using a systematic inspection program
- Ensure that all data are properly archived.

Internal quality control for analytical services will be conducted by the laboratory in accordance to their standard operating procedures, the individual method requirements, and this QAPP. (Refer to Section 5.2.1 for calibration curve analysis and Appendix J for the external lab's QAPP). Before making significant changes to the QAPP or analytical methodology, the laboratory will notify the Project Manager.

Laboratory quality control consists of two distinct components: a laboratory and matrix component. The laboratory component measures the performance of the laboratory analytical process during the sample analyses, while the matrix component measures the effects on the method performance of a specific matrix. Method blanks and laboratory control samples uniquely measure the laboratory component of method performance, while matrix spikes, matrix spike duplicates, laboratory sample duplicates, and surrogate

spikes measure the matrix component of method performance, but also reflect laboratory performance. The following paragraphs discuss the QC samples and parameters to be evaluated to assess the overall laboratory data quality.

### 8.2.1 Holding Time

Holding time reflects the length of time that a sample or sample extract remains representative of the environmental conditions. Depending on the analysis, either one or two holding times will be evaluated. For those analyses that do not include sample extraction, one holding time will be evaluated: the amount of time between sampling and analysis. For analyses that have an extraction prior to analysis, two holding times will be evaluated: 1) the length of time from sampling until extraction, and 2) the length of time from extraction to analysis. Holding times for each analyte are listed in Table 6-1 of Analytical results of samples whose holding times are exceeded are considered quantitatively questionable and may be biased low.

### 8.2.2 Blind Field Replicate and Field Duplicate Samples

Like the field procedures, the analytical procedures will be evaluated by comparing the analytical results of blind field replicates and the field replicate sample pairs. If the reported values for the respective sample pair are similar, the samples are considered to be representative of the environment. A large difference (greater than 40 percent) between the reported values for the sample pair indicates that there may have been a problem during sampling or analysis. Blind field replicate and field duplicate analyses will be used to evaluate precision by calculating the RPD between a blind sample and its associated environmental sample. Section 6.3.3 outlines the procedures for collecting blind field replicate and field duplicate samples.

### 8.2.3 Method Blanks

Method blanks will be used to evaluate representativeness by identifying any contaminants that have been introduced during analysis. Method blanks are generated in the laboratory and consist of ultra-pure water. Method blanks are carried through each processing step necessary for an analytical procedure and are analysed at frequency of one per 20 samples or daily, whatever is more frequent. These blanks measure contamination originating from the laboratory (i.e. water, air, reagents, equipment, and instruments used for analysis), and help in distinguishing low-level field contamination from laboratory contamination. If analytes of interest are found in both the method blank and in associated environmental samples, the data from the associated samples may be considered quantitatively questionable depending on the relative concentrations of contaminants in the method blank and the environmental sample.

#### 8.2.4 Surrogate Spikes

Surrogate spikes will be used to evaluate the accuracy of the analytical method and instrument for CVOC analyses (only the external lab will perform surrogate spikes). During analysis surrogate spike compounds behave similarly to the analytes of interest. Surrogates are added to a few samples and method blanks, including QC samples, prior to extraction or analysis. After the analysis has been completed, the percent recovery of each surrogate spike will be calculated and compared to the established QC limits (see Table 1-1). Good percent recoveries indicate acceptable accuracy during analysis. Poor recoveries indicate that there may have been a problem during analysis (matrix or non-matrix) and that the data may be of questionable value.

#### 8.2.5 Laboratory Control Samples

Laboratory control samples (LCS) will be used to evaluate accuracy and precision. These samples are carried through the same analytical procedures as the environmental samples and are used to evaluate method and analytical procedure performance in the absence of matrix interference. Laboratory control samples are prepared in the laboratory and consist of ultra-pure water that is spiked with specific compounds as outlined in the analytical methods. An LCS sample will be prepared and analysed at a frequency of one per 20 samples, or daily, whichever is more frequent. Accuracy will be evaluated by calculating the percent recovery for each spiked compound and comparing it to the QC limits established by the individual methods (see Appendix H and Appendix I for the UW labs and the external lab). Values within the established QC limits indicate acceptable analytical accuracy. Values outside the QC limits indicate that the data may be questionable. Precision will be evaluated by calculating the RPD of the MS/MSD (performance data) sample pairs, the blind field replicates and the field duplicate sample pairs and comparing the results to laboratory established RPD control limits (see table 1-1 and 1-2).

#### 8.2.6 Matrix Spike and Matrix Spike Duplicate Samples

Results of MS/MSD sample analysis will be used to evaluate accuracy and precision for performance samples only. Unlike LCS, MS/MSD samples are used to assess the influence of the sample matrix (matrix interference) on the analysis. Each MS/MSD sample will be spiked with the compounds specified by the analytical method. To evaluate accuracy the percent recovery for each spiked compound will be calculated and compared to the QC limits established by the method (see the external lab's QAPP, Appendix I). Precision will be evaluated by calculating the RPD between the MS and MSD samples for each spiked analyte. These RPDs will be compared to the QC limits established by laboratory performance (see the external lab's QAPP, Appendix I). Percent recovery and RPD values within the QC limits indicate acceptable precision and accuracy. Values outside the QC limits indicate that there may have been a matrix interference during analysis. The laboratory data validation protocol will be based on precision and accuracy measurement from MS/MSDs. Individual compound recoveries will be compared with acceptance limits. If a matrix spike analyte fails acceptance criteria, the MS/MSD will be

reanalysed and a LCS also will be analysed. For the method to be considered in control, those compounds that failed the matrix spike criteria must be within acceptance limits in the LCS. If, after re-analysis, analytes that failed acceptance criteria in the MS and MSD pass acceptance criteria in the LCS, these analytes may be considered biased due to sample matrix effects.

All samples analysed or prepared in a process batch without an MS and MSD will, at a minimum, have a method blank and LCS. The environmental samples in this batch will be considered in control if more than 80 percent of the target compounds in the LCS are within acceptance limits.

## **9. PERFORMANCE SYSTEM AUDITS**

### **9.1 Field Audits**

Oversight of UW's field procedure will be the direct responsibility of the UW Project Manager, who will review all elements of the QAPP to ensure that the objectives of the experiment are met. In addition to an initial review, the sampling procedures will be reviewed as the field work progresses so that any necessary modifications can be made.

Optional internal audits of the UW's field activities (sampling and measurements) may be conducted by the UW QC Co-ordinator or the co-ordinator's designee. The audits will include examining field measurement records, field equipment calibration records, field sampling records, field instrument operation records, sample collection procedures, sample handling and shipping procedures, and chain-of-custody procedures. Field activities may be audited early in the project to verify that all of the procedures outlined in the FSP and QAPP are being followed. Follow-up audits may be conducted to correct deficiencies, and to verify that QA procedures are maintained throughout the project.

### **9.2 Laboratory Audits**

During the demonstration, system audits will be conducted by reviewing the QC data from the lab. A formal system audit of the external laboratory will not be performed by Rice since ITS is both a Navy and EPA certified lab. The University's Organic Geochemistry laboratory and the Inorganic Geochemistry Lab will also be audited via inspection of the QC data. The University's labs will also be inspected for their procedures on sample receiving, log-in, storage, chain-of-custody documentation, sample preparation and analysis; and instrumentation procedures.

## **10. PREVENTATIVE MAINTENANCE**

### **10.1 Field Equipment**

The field equipment that will be used during this investigation includes an electronic

water-level sounder, pH meter, DO meter, EC and temperature meter. All meters and instruments will be maintained and used according to the manufacturer's directions. Each piece of equipment will be inspected on a regular basis to ensure that the equipment is operational. Any preventative maintenance or repair conducted in the field will be recorded in the field log book.

## **10.2 Laboratory Equipment**

All maintenance performed on laboratory equipment will be documented in the logbook. Receipts from routine maintenance performed by the manufacturer's representative are kept in folders and filed in the laboratory's file cabinets.

In the event of instrument failure, every effort will be made to analyse samples by alternative means within holding times. If the redundancy in equivalent instrumentation is insufficient to handle the affected samples, efforts will be made to secure the same or equivalent analyses at another location.

## **11. DATA ASSESSMENT PROCEDURES**

The quality of the field and analytical data will be evaluated using precision, accuracy, representativeness, completeness, and comparability (PARCC) parameters, which are quantitative and qualitative statements that describe data quality. The PARCC parameters will be used to determine whether the data quality objectives of this investigation have been met by comparing QC sample results and standard procedures with acceptance criteria established for this project. The PARCC parameters that will be used for data evaluation are defined in Section 2.3.

### **11.1 Field Data**

Field measurement data will be assessed by the Project Manager of UW and by the AATDF Representative. The data quality evaluation, in terms of the PARCC parameters, will focus primarily on the laboratory data. However, the field data will be evaluated qualitatively in terms of the PARCC parameters. The following sections discuss how the PARCC parameters will be used to evaluate the field data and field sampling procedures.

#### **11.1.1 Precision**

Sampling precision is affected by the procedures used for sample collection, handling, and transportation. To reduce the variability that may be introduced during sampling, the Sampling and Analysis Plan and section 4.0 outlines the standard sampling, handling, and shipping procedures that will be used for each sampling program. The use of these procedures should minimize variability in the sampling process.

In addition, the results of blind field replicates and field duplicates sample analyses also will be used to evaluate sampling precision. The RPD will be calculated for each blind

field replicate sample pair. Although the results of blind field replicate sample analyses also reflect the variability associated with analytical procedures, low RPD values are an indication that consistent sampling techniques were used for sample collection.

#### 11.1.2 Accuracy

Although there is no way to quantitatively measure the accuracy of the field program using percent recovery, some aspects of accuracy can be assessed, such as comparing the length of the water-level probe to another measuring tape of known length and proper calibration of the field instruments.

#### 11.1.3 Representativeness

The representativeness of the field data is determined by the design of the data collection procedures. The sampling and field measurement procedures to be used are based on existing analytical data, hydrogeology, and the physical setting of Site 1. However, due to the spatial heterogeneity of the aquifer, there is little ability to demonstrate absolute representativeness of the samples collected at a well.

Representativeness of the field sampling procedures and the field measurements will be evaluated by comparing the sampling and measurement procedures used in the field to the procedures outlined in the Sampling and Analysis Plan and this QAPP. In addition, the results of equipment blank samples will be used to evaluate the representativeness of field sampling procedures. Contaminants detected in equipment blanks are indications that the decontamination procedures are not completely effective, and that contaminants detected at specific sites may be attributable to cross-contamination rather than the environment.

#### 11.1.4 Comparability

The comparability of the field sampling procedures and field measurement data will be evaluated by comparing them to previous sampling rounds.

#### 11.1.5 Completeness

Completeness of the field program will be evaluated to ensure that the appropriate number of samples were collected for analysis, and that field data of the type and quantity outlined in the Sampling and Analysis Plan were collected. Completeness of the field investigations will be evaluated by comparing the actual number of samples and the actual quantity of data that were collected to the requirements outlined in the Sampling and Analysis Plan.

### **11.2 Laboratory Data**

The laboratory data will be assessed by the Project Manager of UW and will be based on the assumption that the sample was collected, handled, and analysed according to the Sampling and Analysis Plan and this QAPP. The Project Manager will conduct a

systematic review of the data for compliance with the QC criteria established in the QAPP, and will identify any data omissions or data that do not meet the quality control criteria. The reviewer also will interact with both laboratories to correct any data deficiencies. Decisions to repeat sample collection or analyses will be made by the UW or Rice based on the extent of the data deficiencies and their importance in the overall content of the project. Results of the data assessment will be presented in an appendix of the final report scheduled to summarize the results of this investigation.

As discussed above, PARCC parameters will be used to evaluate the quality of analytical data and determine whether the data quality objectives of the project have been met. To assess the quality of the analytical data, the results of the QC sample analyses will be evaluated using quality control limits established by the analytical methods used for analysis, or by past laboratory performance. Results of the quality control sample evaluation then will be expressed in terms of the PARCC parameters and used to assess the quality of the analytical data.

The quality control samples that will be used to evaluate the analytical data for this program include trip blanks, equipment blanks, blind field replicate samples, field duplicates samples, method blanks, surrogate spikes (when applicable), laboratory control samples (when applicable) and matrix spike/matrix spike duplicate samples. The following sections describe the criteria that will be used to evaluate the laboratory data.

#### 11.2.1 Precision

Analytical precision is determined by analysing field duplicates or replicates submitted "blind" to the laboratory, and MS/MSD samples (for performance data only). Relative percent difference is calculated between the sample pairs and compared with control limit acceptance criteria. The data quality objectives for precision during this program are based on laboratory established control limits, which are specific to each analyte (see table 1-1 and 1-2).

#### 11.2.2 Accuracy

Accuracy is a quantitative measure of the bias of a method or the level of agreement between a measurement of a known true value. Laboratory accuracy will be evaluated using the results for surrogate spike, MS/MSD, and LCS/LCSD sample analyses for performance data and surrogate spike and LCS/LCSD for treatment analyses.. As with precision, the accuracy objectives for the data are based on laboratory established limits, and vary with the specific analyte.

#### 11.2.3 Representativeness

Representativeness is a qualitative parameter that evaluates whether or not the data represent the actual environmental conditions. Representativeness will be evaluated by analysis of laboratory method and equipment blanks, and blind duplicate or replicate

samples. Representativeness is also evaluated using holding-time criteria, which reflects the length of time that a sample remains representative of the environmental conditions after sample collection. Holding times are compared to standard method-specific holding times accepted by the EPA. All holding times within the acceptance criteria are considered representative. Those holding times outside the EPA acceptance criteria are qualitatively evaluated to determine the effect on sample representativeness.

#### 11.2.4 Comparability

Comparability is a qualitative expression of the confidence with which one data set can be compared to another. Comparability is maximized through the use of standard analytical method and units of measurement.

#### 11.2.5 Completeness

Completeness is expressed as a percentage and is defined as the number of valid samples relative to the total number of samples gathered during the sampling programs.

## **12. CORRECTIVE ACTIONS**

### **12.1 Field Programs**

Sample collectors will be responsible for documenting and reporting all suspected technical and QA non-conformances, and suspected deficiencies during any field activity. The non-conformances and/or deficiencies will be documented in the field log book and reported to UW. If the problem is associated with field measurements or sampling equipment, the sample collectors will take the appropriate steps to correct the problem. Typical procedures to correct problems include the following:

- Repeating the measurement to check for error
- Making sure the meters or instruments are adjusted properly for the ambient conditions, such as temperature
- Checking or replacing batteries
- Recharging batteries
- Recalibrating the instruments
- Replacing the meters or instruments used to measure field parameters
- Stopping work until the problem is corrected

If a non-conformance or problem requires a major adjustment to the field procedures as outlined in the Sampling and Analysis Plan, the Project Manager from UW, will be responsible for initiating corrective actions. The Project Manager will be responsible for the following:

- Evaluating the reported non-conformance
- Controlling additional work on non-conforming items
- Determining the appropriate corrective actions
- Maintaining a log of all non-conformance and corrective actions
- Ensuring that explanations of non-conformance and corrective actions is included in an appendix of the final report for this investigation

The Project Manager will ensure that no additional work that is dependent on the nonconforming activity is performed until the appropriate corrective actions are completed.

## **12.2 Laboratory Analysis**

Corrective actions are required whenever unreliable analytical results prevent the quality control criteria as specified by the method or the laboratory QAPP from being met. The corrective actions will be undertaken if one of the following occurs:

- QC data are outside the acceptance windows for precision and accuracy
- Some data points are identified as outlying points on the breakthrough curves and time series
- Blanks contain contaminants above acceptance levels (see Appendix H and I for the external's lab QAPP and UW's analytical procedures respectively).
- Undesirable trends are detected for spike recoveries (or spike recoveries are outside the QC limits) or RADS between duplicate analyses are consistently outside QC limits
- There are unusual changes of detection limits during analysis
- Deficiencies are detected during QA audits

Corrective actions are primarily handled at the bench level by the analyst who reviews the sample preparation or extraction procedures, and performs the instrument calibration and analysis. If the problem persists or its cause cannot be identified, the matter will be referred to the project manager or QA officer (of the specific lab) for further investigation. Once resolved, full documentation of the corrective action procedure will be filed with the QA officer of the specific lab. A summary of the corrective actions will be included in the data package submitted to UW.

## **13. QUALITY ASSURANCE REPORTS**

With the analytical data reported from calibration curve analysis, each analysis has a quality control number attached to it. These numbers will remain together in the project database. The majority of the data will be analysed this way. In those cases where calibration curves are not used, only analyses for which breaches in the quality control are

suspected (based on replicates and blanks) will trigger a quality control report to the project manager.

**APPENDIX L**

**ENVIRONMENTAL PROTECTION PLAN**

## Environmental Protection Plan

There is one building located in the southern part of Site 1, directly south of where the Demonstration Project will be installed. Overall, the site surface is mostly fill materials with a sparse presence of grasses and weeds. There does not appear to be any burrows, or nests, or scat to indicate the presence of any on-site fauna. Because this is a demonstration project site, it will be up to the discretion of the EPA-West to restore and/or reseed this area.

Before work activities commence, a standard six-foot cyclone fence will be installed around the perimeter of Site 13 and an exclusionary zone will be identified and marked around the entrance of the site to protect project workers as well as people walking in this area. A small shed to house oxygen sparging equipment, extraction pumps, electrical outlets, etc. will be erected near the down gradient part of the treatment gate.

Fugitive dust emissions during grading operations will be controlled by light watering of affect areas as directed by the construction site superintendent or as directed by the Resident Officer in Charge of Construction (ROICC) based upon wind velocity and site observations. To construct the Demonstration Project, the Contractor will excavate native materials from the sheet pile box and dewater the box if necessary. The box will be braced as required typically using structural steel. Native material will be screened, segregated, sampled and stored on site until appropriate off site disposal is undertaken. Disposal of all native material will be the responsibility of the Contractor who will follow all appropriate state and local laws. The wastewater generated from the excavation will be analysed, stored temporarily on site and then, if appropriate will be sent to the Industrial Wastewater Treatment Plant (IWTP), located on base. These arrangements will be made by the Contractor.

Before conducting any invasive work, all underground utilities will be located, and the area cleared by an underground utilities locating service. The locations of all utilities will be clearly marked with either paint, caution flags, or tape on stakes or temporary barricades, as appropriate, to the activities at the location and the element of risk. Existing water, sanitary, and storm sewer lines will not be removed or rerouted nor will any associated vaults or drains be removed or interfered with.

The area will be surveyed for radioactive materials prior to moving in equipment. The survey will be conducted by walking a series of traverses correlating to a 4-foot-centre grid pattern on the area to be surveyed using a radiation survey meter and a scintillation counter and a sodium-iodide crystal detector.

Groundwater monitoring wells at the site will be protected during the Demonstration Project. As necessary, each groundwater monitoring well will be completely sealed with plastic, or a corrugated metal cover will be placed over the well head.

AS PER TABLE OF CONTENTS, PLATES 1 AND 2  
ARE NOT INCLUDED IN THIS DOCUMENT.  
PLATES 1 AND 2 ARE THE SAME AS VERSION 0.

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