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**Virginia Wood Preserving Site
Richmond, Virginia**

**Phase II Work Plan and
Quality Assurance Project Plan**

Volume II - Appendices A through E

Prepared for:

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AR301955

APPENDIX A
Additional Standard Operating Procedures

AR301956

SOP NUMBER: C.I.1

DATE: June 1990

TITLE: Jar Headspace Test

SCOPE: This operating procedure describes techniques for screening soil samples in the field to obtain a relative indication of the general volatile organic compound concentrations present.

OBJECTIVES: Rapid screening of soil for volatile organic compounds

EQUIPMENT:

- PID (see SOP B.IV.1 in WP/QAPjP)
- Screwtop glass jar (minimum 8 oz.)
- Aluminum foil

PROCEDURE:

1. Collect soil sample from split spoon after completing description. For samples to be chemically analyzed, the residue will be used in this test. (These materials must be disturbed as little as possible.)
2. Fill two clean glass jars halfway with soil, quickly cover with aluminum foil, apply screw cap (minimum 8-oz. jar).
3. Shake jars for 15 seconds, wait at least 10 minutes and shake again for 15 seconds.
4. Successively remove screw caps and insert probe of PID through foil in each jar.
5. Record highest reading.

SOP NUMBER: C.I.2

DATE: June 1990

TITLE: Shelby Tube Sampling (based on ASTM Method 1587)

SCOPE: This operating procedure describes the procedures involved in collecting undisturbed soil samples using a soil sampling shelly tube based on ASTM Method 1587.

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in collecting soil samples using a shelly tube.
- Ensure that the sample collected will be undisturbed and representative of actual existing conditions in the field.
- Provide consistency in field methods used to collect soil samples with a shelly tube.

EQUIPMENT:

- Drilling rig and drilling equipment
- Shelby tube(s)
- Shipping containers and labels
- Allen wrenches
- Tape measure
- Preserving materials (wax, foil, etc.)
- Field log.

PRELIMINARY TO OPERATION:

1. Examine the shelly tube for cleanliness, and check for defects. This should include making sure that set screws that hold the shelly tube in place are intact.
2. Verify that the soil being sampled is clayey, or loose silts, or sands. Shelby tubes should not be used in dense sand, gravelly material, or rock.

PROCEDURE:

1. Ground control for sample location shall be within ± 2 feet of the identified location or consistent with data quality objectives.
2. Clean the bottom of the hole in which the shelby tube will be inserted to the sampling elevation.
3. Attach the shelby tube to the lower end of the drill rods and secure set screws with allen wrenches.
4. Lower the shelby tube to the sampling elevation at the bottom of the hole with the drilling apparatus.
5. Mark off on the drill rods an interval length equivalent to the length of the shelby tube.
6. Push the shelby tube into the ground with a continuous motion without impact or twisting. This is typically performed using the hydraulic mechanism on the drilling rig.
7. Push the tube into the ground until the uppermost mark on the drill stem is in exact position as the lower mark was before the tube was lowered.
8. If soils are so hard that the shelby tube cannot be pushed, a driving hammer may be used to advance the sampler. If this is the case, record the weight, height, and number of blows in the field log.
9. When the tube has been advanced to the appropriate depth, twist the tube at least two revolutions to shear off at the bottom. Raise the sample.
10. Disconnect the shelby tube and sample with the set screws.
11. If the sample is to be extracted from the shelby tube in the lab, remove about 1/4 inch of the soil from

each end of the tube, wipe the tube end (inside and out) with a clean towel, and seal the ends of the tube with paraffin wax. Place a plastic shipping cap over each end of the tube and wrap with plastic tape.

12. If the sample is to be extracted from the shelby tube at the site, seal the extracted sample in a glass jar and decontaminate the tube before collecting next sample.
13. Label the samples with the shelby tube soil sample label and record sample characteristics, including color and texture, in the field log.

SOP NUMBER: C.I.3

DATE: December 1990

TITLE: Determination of effective porosity and intrinsic permeability

SCOPE: This operating procedure describes the laboratory methodology used in determination of effective pore volume (California State Water Resources Control Board) and intrinsic permeability (EPA 9100) from undisturbed soil samples collected using a Shelby Tube (see SOP C.I.2)

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the laboratory.
- Identify the mechanisms, materials, and procedure that will ensure the proper testing of soils for determination of effective porosity and intrinsic permeability.

EQUIPMENT: See Attachment

PRELIMINARY
TO OPERATION: See Attachment

PROCEDURE: See Attachment

AR301961

METHOD FOR LABORATORY DETERMINATION OF EFFECTIVE PORE VOLUME¹

Step 6: Characterize the Clay Liner

Guidance

Characterization should include the determination of effective pore volume and permeability of the clay liner.

Method

Characterization of a clay liner will not, in itself, reveal if a liner has all the appropriate properties to prevent failure. This characterization can, however, establish the baseline permeability and effective pore volume values with a standard aqueous leachate (0.01 N CaSO_4) and nonattenuated ion (bromide), respectively. These baseline values can be compared with the values obtained with the leachates of a waste. Suitable methods for analyzing soil physical properties are available in the latest edition of ASTM Standards (part 19: Natural Building Stones; Soil and Rock), in Black (1963), or in most soil testing manuals. Determination of bromide breakthrough can be accomplished by using a bromide selective electrode to analyze permeameter outflow. The following information should be determined for the clay liner.

- a. Particle size distribution and clay mineralogy
 1. _____ percent Sand ($> 50 \mu\text{m}$, dry wt. basis)
 2. _____ percent Silt ($50 - 2.0 \mu\text{m}$, dry wt. basis)
 3. _____ percent Clay ($< 2.0 \mu\text{m}$, dry wt. basis)
 4. _____ percent of Clay that is coarse ($2.0 - 0.2 \mu\text{m}$, dry wt. basis)
 5. Predominant coarse Clay minerals
_____ percent _____
_____ percent _____
_____ percent _____
 6. _____ percent of Clay that is fine ($0.2 \mu\text{m}$, dry wt. basis)
 7. Predominant fine Clay minerals
_____ percent _____
_____ percent _____
_____ percent _____
- b. Physical properties
 1. _____ g cm^{-3} particle density
 2. _____ percent in-place (as compacted) water content (dry wt. basis)
 3. _____ g cm^{-3} in-place (as compacted) density (dry wt. basis)
 4. _____ percent pore space (percent of total liner volume)
 5. Permeability to an aqueous solution of 0.02 N CaSO_4 or CaCl_2 after percolation of:
0.5 pore volume _____ cm/sec
1.0 pore volume _____ cm/sec
1.5 pore volume _____ cm/sec
2.0 pore volume _____ cm/sec
 6. Pore volume values for Bromide breakthrough
_____ percent Bromide at 0.1 pore volume
_____ percent Bromide at 0.2 pore volume
_____ percent Bromide at 0.3 pore volume
_____ percent Bromide at 0.4 pore volume
_____ percent Bromide at 0.5 pore volume
_____ percent Bromide at 0.6 pore volume
_____ percent Bromide at 0.7 pore volume
_____ percent Bromide at 0.8 pore volume
_____ percent Bromide at 0.9 pore volume
_____ percent Bromide at 1.0 pore volume

The permeability of the clay liner should first be evaluated using a standard leachate (such as 0.01 N CaSO_4 or CaCl_2). After the permeability has stabilized, the effective pore volume can be estimated by spiking the standard leachate with bromide and monitoring the breakthrough of bromide in the permeameter outflow. Assuming that a clay liner is presaturated with clean water, nonattenuated leachate constituents (e.g., bromide) will be present in the outflow from the permeameter after passage of leachate equal to approximately 50 percent of the volume represented by one effective pore volume.

¹Methodology Contained in California Code of Regulations, Title 23, Waters, Chapter 3-State Water Resources Control Board, Article 10, Section 2601.

AR301962

METHOD 9100

SATURATED HYDRAULIC CONDUCTIVITY, SATURATED LEACHATE CONDUCTIVITY, AND INTRINSIC PERMEABILITY

1.0 INTRODUCTION

1.1 Scope and Application: This section presents methods available to hydrogeologists and geotechnical engineers for determining the saturated hydraulic conductivity of earth materials and conductivity of soil liners to leachate, as outlined by the Part 264 permitting rules for hazardous-waste disposal facilities. In addition, a general technique to determine intrinsic permeability is provided. A cross reference between the applicable part of the RCRA Guidance Documents and associated Part 264 Standards and these test methods is provided by Table A.

1.1.1 Part 264 Subpart F establishes standards for ground water quality monitoring and environmental performance. To demonstrate compliance with these standards, a permit applicant must have knowledge of certain aspects of the hydrogeology at the disposal facility, such as hydraulic conductivity, in order to determine the compliance point and monitoring well locations and in order to develop remedial action plans when necessary.

1.1.2 In this report, the laboratory and field methods that are considered the most appropriate to meeting the requirements of Part 264 are given in sufficient detail to provide an experienced hydrogeologist or geotechnical engineer with the methodology required to conduct the tests. Additional laboratory and field methods that may be applicable under certain conditions are included by providing references to standard texts and scientific journals.

1.1.3 Included in this report are descriptions of field methods considered appropriate for estimating saturated hydraulic conductivity by single well or borehole tests. The determination of hydraulic conductivity by pumping or injection tests is not included because the latter are considered appropriate for well field design purposes but may not be appropriate for economically evaluating hydraulic conductivity for the purposes set forth in Part 264 Subpart F.

1.1.4 EPA is not including methods for determining unsaturated hydraulic conductivity at this time because the Part 264 permitting standards do not require such determinations.

1.2 Definitions: This section provides definitions of terms used in the remainder of this report. These definitions are taken from U.S. Government publications when possible.

TABLE A

HYDRAULIC AND LINER CONDUCTIVITY DETERMINATION
METHODS FOR SURFACE IMPOUNDMENT,
WASTE PILE, AND LANDFILL COMPONENTS, AS CITED
IN RCRA GUIDANCE DOCUMENTS AND DESCRIBED IN SW-846

Surface Impoundments	Guidance Cite ¹ Associated Regulation	Corresponding SW-846 Section
Soil liner hydraulic conductivity	Guidance section D(2)(b)(1) and D(2)(c)(1)/Section 264.221(a), (b)	2.0
Soil liner leachate conductivity	Guidance section D(2)(b)(2) and D(2)(c)(2)	2.11
Leak detection	Guidance section C(2)(a)/Section 264.222	2.0
Final cover drain layer	Guidance section E(2)(d)(1) Section 264.228	2.0
Final cover low permeability layer	Guidance section E(2)(e)(2)(A)/Section 264.228	2.0
General hydrogeologic site investigation	264 subpart F	3.0

¹ RCRA Guidance Document: Surface Impoundments, Liner Systems, Final Cover, and Freeboard Control. Issued July, 1982.

(continued on next page)

TABLE A (continued)

Waste Piles	Guidance Cite ² Associated Regulation	Corresponding SW-846 Section
Soil liner hydraulic conductivity	Guidance section D(2)(b)(i) and D(2)(c)(i) / Section 264.251(a)(1)	2.0
Soil liner leachate conductivity	Guidance section D(2)(b)(ii) and D(2)(c)(ii)	2.11
Leak detection system	Guidance section C(2)(a) / Section 264.252(a)	2.0
Leachate collection and renewal system	Guidance section C(2)(a) / Section 264.251(a)(2)	2.0
General hydrogeologic site investigation	264 subpart F	3.0

² RCRA Guidance Document: Waste Pile Design, Liner Systems.
Issued July, 1982.

(continued on next page)

TABLE A (continued)

Landfills	Guidance Cite ³ Associated Regulation	Corresponding SW-846 Section
Soil liner hydraulic conductivity	Guidance section D(2)(b)(1)/ Section 264.301(a)(1)	2.0
Soil liner leachate conductivity	Guidance section D(2)(b)(2)	2.11
Leak detection system	Guidance section C(2)(a)/ Section 264.302(a)(3)	2.0
Leachate collection and removal system	Guidance section C(2)(a)/ Section 264.301(a)(2)	2.0
Final cover drain layer	Guidance section E(2)(d)(1)/ Section 264.310(a)(b)	2.0
Final cover low permeability layer	Guidance section E(2)(e)(2)(A) Section 264.310(a)(b)	2.0
General hydrogeologic site investigation	264 subpart F	3.0

³ RCRA Guidance Document:
Issued July, 1982.

Landfill Design, Liner Systems and Final Cover.

1.2.1 Units: This report uses consistent units in all equations. The symbols used are:

Length = L,
Mass = M, and
Time = T.

1.2.2 Fluid potential or head (h): A measure of the potential energy required to move fluid from a point in the porous medium to a reference point. For virtually all situations expected to be found in disposal sites and in ground water systems, h is defined by the following equation:

$$h = h_p + h_z \quad (1)$$

where:

h is the total fluid potential, expressed as a height of fluid above a reference datum, L;

h_p , the pressure potential caused by the weight of fluid above the point in question, L, is defined by $h_p = P/\rho g$,

where:

P is the fluid pressure at the point in question, $ML^{-1}T^{-2}$,

ρ is the fluid density at the prevailing temperature, ML^{-3} ,
and

g is the acceleration of gravity, LT^{-2} ; and

h_z is the height of the point in question above the reference datum, L.

By knowing h_p and h_z at two points along a flow path and by knowing the distance between these points, the fluid potential gradient can be determined.

1.2.3 Hydraulic potential or head: The fluid potential when water is the fluid.

1.2.4 Hydraulic conductivity: The fluid potential when water is the fluid. The generic term, fluid conductivity, is discussed below in 1.2.5.

1.2.5 Fluid conductivity (K): Defined as the volume of fluid at the prevailing density and dynamic viscosity that will move in a unit time under a unit fluid potential gradient through a unit area measured at right angles to the direction of flow. It is a property of both the fluid and the porous medium as shown by the following equation:

$$K = \frac{k\rho g}{u};$$

(2)

where:

K is the fluid conductivity, LT^{-1} ;

k is the intrinsic permeability, a property of the porous medium alone, L^2 ; and

u is the dynamic viscosity of the fluid at the prevailing temperature, $ML^{-1}T^{-1}$.

The fluid conductivity of a porous material is also defined by Darcy's law, which states that the fluid flux (q) through a porous medium is proportional to the first power of the fluid potential across the unit area:

$$q = \frac{Q}{A} = -KI \quad (3)$$

where:

q = the specific fluid flux, LT^{-1} ,

Q is the volumetric fluid flux, L^3T^{-1} ,

A is the cross-sectional area, L^2 , and

I is the fluid potential gradient, L^0 .

Darcy's law provides the basis for all methods used to determine hydraulic conductivity in this report. The range of validity of Darcy's law is discussed in Section 1.5 (Lohman, 1972).

1.2.6 Leachate conductivity: The fluid conductivity when leachate is the fluid.

1.2.7 Aquifer: A geologic formation, group of formations, or part of a formation capable of yielding a significant amount of ground water to wells or springs (40 CFR 260.10).

1.2.8 Confining layer: By strict definition, a body of impermeable material stratigraphically adjacent to one or more aquifers. In nature, however, its hydraulic conductivity may range from nearly zero to some value distinctly lower than that of the aquifer. Its conductivity relative to that of the aquifer it confines should be specified or indicated by a suitable modifier, such as "slightly permeable" or "moderately permeable" (Lohman, 1972).

1.2.9 Transmissivity, T [L^2, T^{-1}]: The rate at which water of the prevailing kinematic viscosity is transmitted through a unit width of the aquifer under a unit hydraulic gradient. Although spoken of as a

property of the aquifer, the term also includes the saturated thickness of the aquifer and the properties of the fluid. It is equal to an integration of the hydraulic conductivities across the saturated part of the aquifer perpendicular to the flow paths (Lohman, 1972).

1.3 Temperature and viscosity corrections: By using Equation (2), corrections to conditions different from those prevailing during the test can be made. Two types of corrections can commonly be made: a correction for a temperature that varies from the test temperature, and a correction for fluids other than that used for the test. The temperature correction is defined by:

$$K_f = \frac{K_t u_t \rho_f}{u_f \rho_t} \quad (4)$$

where:

the subscript f refers to field conditions, and

the subscript t refers to test conditions.

Most temperature corrections are necessary because of the dependence of viscosity on temperature. Fluid density variations caused by temperature changes are usually very small for most liquids. The temperature correction for water can be significant. Equation (4) can also be used to determine hydraulic conductivity if fluids other than water are used. It is assumed, however, when using Equation (4) that the fluids used do not alter the intrinsic permeability of the porous medium during the test. Experimental evidence shows that this alteration does occur with a wide range of organic solvents (Anderson and Brown, 1981). Consequently, it is recommended that tests be run using fluids, such as leachates, that might occur at each particular site. Special considerations for using non-aqueous fluids are given in Section 3.3 of this report.

1.4 Intrinsic permeability (k): Rearrangement of Equation 2 results in a definition of intrinsic permeability:

$$k = \frac{Ku}{\rho g} \quad (5)$$

Since this is a property of the medium alone, if fluid properties change, the fluid conductivity must also change to keep the intrinsic permeability a constant. By using measured fluid conductivity, and values of viscosity and density for the fluid at the test temperature, intrinsic permeability can be determined.

1.5 Range of validity of Darcy's law: Determination of fluid conductivities using both laboratory and field methods requires assuming the validity of Darcy's law. Experimental evidence has shown that deviations from the linear dependence of fluid flux on potential gradient exist for both extremely low and extremely high gradients (Hillel, 1971; Freeze and Cherry, 1979). The lower limits are the result of the existence of threshold

gradients required to initiate flow (Swartzendruber, 1962). The upper limits to the validity of Darcy's law can be estimated by the requirements that the Reynolds number, Re , in most cases be kept below 10 (Bear, 1972). The Reynolds number is defined by:

$$Re = \frac{\rho q d}{\mu} \quad (6)$$

where:

d is some characteristic dimension of the system, often represented by the median grain size diameter, D_{50} , (Bouwer, 1978), and

q is the fluid flux per unit area, LT^{-1} .

For most field situations, the Reynolds number is less than one, and Darcy's law is valid. However, for laboratory tests it may be possible to exceed the range of validity by the imposition of high potential gradients. A rough check on acceptable gradients can be made by substituting Darcy's law in Equation (6) and using an upper limit of 10 for Re :

$$I \leq \frac{10\mu}{\rho K D_{50}} \quad (7)$$

where:

K is the approximate value of fluid conductivity determined at gradient I .

A more correct check on the validity of Darcy's law or the range of gradients used to determine fluid conductivity is performed by measuring the conductivity at three different gradients. If a plot of fluid flux versus gradient is linear, Darcy's law can be considered to be valid for the test conditions.

1.6 Method Classification: This report classifies methods of determining fluid conductivity into two divisions: laboratory and field methods. Ideally, and whenever possible, compliance with Part 264 disposal facility requirements should be evaluated by using field methods that test the materials under in-situ conditions. Field methods can usually provide more representative values than laboratory methods because they test a larger volume of material, thus integrating the effects of macrostructure and heterogeneities. However, field methods presently available to determine the conductivity of compacted fine-grained materials in reasonable times require the tested interval to be below a water table or to be fairly thick, or require excavation of the material to be tested at some point in the test. The integrity of liners and covers should not be compromised by the installation of boreholes or piezometers required for the tests. These restrictions generally lead to the requirement that the fluid conductivity of liner and cover materials must be determined in the laboratory. The transfer value of laboratory data to field conditions can be maximized for liners and covers because it is possible to reconstruct relatively accurately the desired

field conditions in the laboratory. However, field conditions that would alter the values determined in the laboratory need to be addressed in permit applications. These conditions include those that would increase conductivity by the formation of microcracks and channels by repeated wetting and drying, and by the penetration of roots.

1.6.1 Laboratory methods are categorized in Section 2.0 by the methods used to apply the fluid potential gradient across the sample. The discussion of the theory, measurement, and computations for tests run under constant and falling-head conditions is followed by a detailed discussion of tests using specific types of laboratory apparatus and the applicability of these tests to remolded compacted, fine-grained uncompacted, and coarse-grained porous media. Section 2.3 provides a discussion of the special considerations for conducting laboratory tests using non-aqueous permeants. Section 2.10 gives a discussion of the sources of error and guidance for establishing the precision of laboratory tests. Laboratory methods may be necessary to measure vertical fluid conductivity. Values from field tests reflect effects of horizontal and vertical conductivity.

1.6.2 Field methods are discussed in Section 3.0 and are limited to those requiring a single bore hole or piezometer. Methods requiring multiple bore holes or piezometers and areal methods are included by reference. Because of the difficulties in determining fluid conductivity of in-place liner and cap materials under field conditions without damaging their integrity, the use of field methods for fine-grained materials will be generally restricted to naturally occurring materials that may serve as a barrier to fluid movement. Additional field methods are referenced that allow determination of saturated hydraulic conductivity of the unsaturated materials above the shallowest water table. General methods for fractured media are given in Section 3.8. A discussion of the important considerations in well installation, construction, and development is included as an introduction to Section 3.0.

2.0 LABORATORY METHODS

2.1 Sample collection for laboratory method: To assure that a reasonable assessment is made of field conditions at a disposal site, a site investigation plan should be developed to direct sampling and analysis. This plan generally requires the professional judgement of an experienced hydrogeologist or geotechnical engineer. General guidance is provided for plan development in the Guidance Manual for Preparation of a Part 264 Land Disposal Facility Permit Application (EPA, in press). The points listed below should be followed:

- o The hydraulic conductivity of a soil liner should be determined either from samples that are processed to simulate the actual liner, or from an undisturbed sample of the complete liner.

- o To obtain undisturbed samples, the thin-walled tube sampling method (ASTM Method # D1587-74) or a similar method may be used. Samples representative of each lift of the liner should be obtained, and used in the analyses. If actual undisturbed samples are not used, the soil used in liner construction must be processed to represent accurately the liner's initial water content and bulk density. The method described in Section 2.7.3 or ASTM Method #D698-70 (ASTM, 1978) can be used for this purpose.
- o For purpose of the general site investigation, the general techniques presented in ASTM method #D420-69 (ASTM, 1978) should be followed. This reference establishes practices for soil and rock investigation and sampling, and incorporates various detailed ASTM procedures for investigation, sampling, and material classification.

2.2 Constant-head methods: The constant-head method is the simplest method of determining hydraulic conductivity of saturated soil samples. The concept of the constant-head method is schematically illustrated in Figure 1. The inflow of fluid is maintained at a constant head (h) above a datum and outflow (Q) is measured as a function of time (t). Using Darcy's law, the hydraulic conductivity can be determined using the following equation after the outflow rate has become constant:

$$K = \frac{QL}{hA}, \quad \frac{Q}{A} = K \frac{h}{L} \quad (8)$$

where:

K = hydraulic conductivity, LT⁻¹;

L = length of sample, L;

A = cross-sectional area of sample, L²;

Q = outflow rate, L³T⁻¹; and

h = fluid head difference across the sample, L.

Constant-head methods should be restricted to tests on media having high fluid conductivity.

2.3 Falling-head methods: A schematic diagram of the apparatus for the falling-head method is shown in Figure 2. The head of inflow fluid decreases from h₁ to h₂ as a function of time (t) in a standpipe directly connected to the specimen. The fluid head at the outflow is maintained constant. The quantity of outflow can be measured as well as the quantity of inflow. For the setup shown in Figure 2a, the hydraulic conductivity can be determined using the following equation:

$$K = \frac{2.3 a l}{A t} \log_{10} \frac{h_0}{h_1}, \quad (9)$$

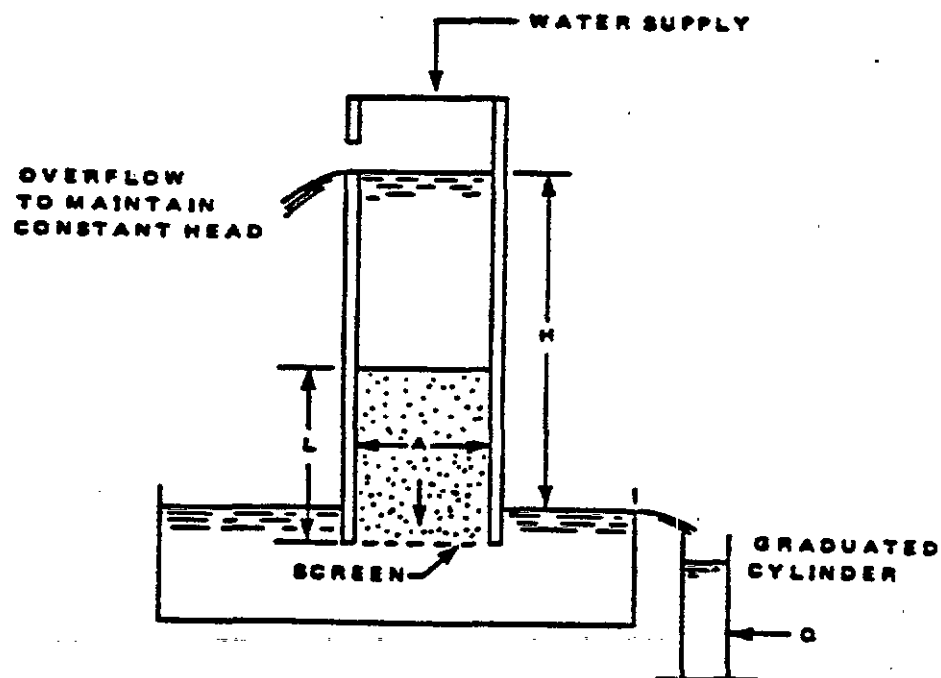


Figure 1.--Principle of the constant head method

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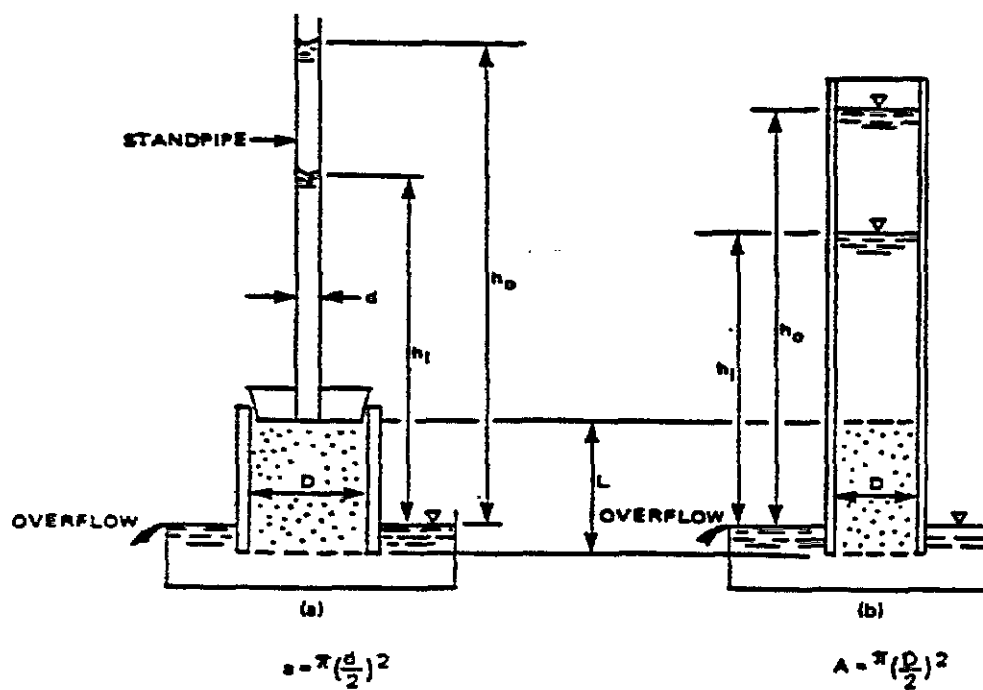


Figure 2.--Principle of the falling head method
using a small (a) and large (b) standpipe.

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where:

a = the cross-sectional area of the standpipe, L^2 ;

A = the cross-sectional area of the specimen, L^2 ;

L = the length of the specimen, L ; and

t = elapsed time from t_1 to t_2 , T .

For the setup in Figure 2b, the term a/A in Equation (9) is replaced by 1.0. Generally, falling-head methods are applicable to fine-grained soils because the testing time can be accelerated.

2.4 General test considerations:

2.4.1 Fluid supplies to be used: For determining hydraulic conductivity and leachate conductivity, the supplies of permeant fluid used should be de-aired. Air coming out of solution in the sample can significantly reduce the measured fluid conductivity. Deairing can be achieved by boiling the water supply under a vacuum, bubbling helium gas through the supply, or both.

2.4.1.1 Significant reductions in hydraulic conductivity can also occur in the growth and multiplication of microorganisms present in the sample. If it is desirable to prevent such growth, a bactericide or fungicide, such as 2000 ppm formaldehyde or 1000 ppm phenol (Olsen and Daniel, 1981), can be added to the fluid supply.

2.4.1.1 Fluid used for determining hydraulic conductivity in the laboratory should never be distilled water. Native ground water from the aquifer underlying the sampled area or water prepared to simulate the native ground water chemistry should be used.

2.4.2 Pressure and Fluid Potential Measurement: The equations in this report are all dimensionally correct; that is, any consistent set of units may be used for length, mass, and time. Consequently, measurements of pressure and/or fluid potential using pressure gages and manometers must be reduced to the consistent units used before applying either Equation 8 or 9. Pressures or potentials should be measured to within a few tenths of one percent of the gradient applied across the sample.

2.5 Constant-head test with conventional permeameter:

2.5.1 Applicability: This method covers the determination of the hydraulic conductivity of soils by a constant-head method using a conventional permeameter. This method is recommended for disturbed coarse-grained soils. If this method is to be used for fine-grained soils, the testing time may be prohibitively long. This method was taken from the Engineering and Design, Laboratory Soils Testing Manual (U.S. Army, 1980). It parallels ASTM Method D2434-68 (ASTM, 1978). The ASTM

method gives extensive discussion of sample preparation and applicability and should be reviewed before conducting constant-head tests. Lambe (1951) provides additional information on sample preparation and equipment procedures.

2.5.2 Apparatus: The apparatus is shown schematically in Figure 3. It consists of the following:

1. A permeameter cylinder having a diameter at least 8 times the diameter of the largest particle of the material to be tested;
2. Constant-head filter tank;
3. Perforated metal disks and circular wire to support the sample;
4. Filter materials such as Ottawa sand, coarse sand, and gravel of various gradations;
5. Manometers connected to the top and bottom of the sample;
6. Graduated cylinder, 100-mL capacity;
7. Thermometer;
8. Stop watch;
9. Deaired water;
10. Balance sensitive to 0.1 gram; and
11. Drying oven.

2.5.3 Sample preparation:

1. Oven-dry the sample. Allow it to cool, and weigh to the nearest 0.1 g. Record the oven-dry weight of material. The amount of material should be sufficient to provide a specimen in the permeameter having a minimum length of about one to two times the diameter of the specimen.
2. Place a wire screen, with openings small enough to retain the specimen, over a perforated disk near the bottom of the permeameter above the inlet. The screen opening should be approximately equal to the 10 percent size of the specimen.
3. Allow deaired water to enter the water inlet of the permeameter to a height of about 1/2 in. above the bottom of the screen, taking care that no air bubbles are trapped under the screen.

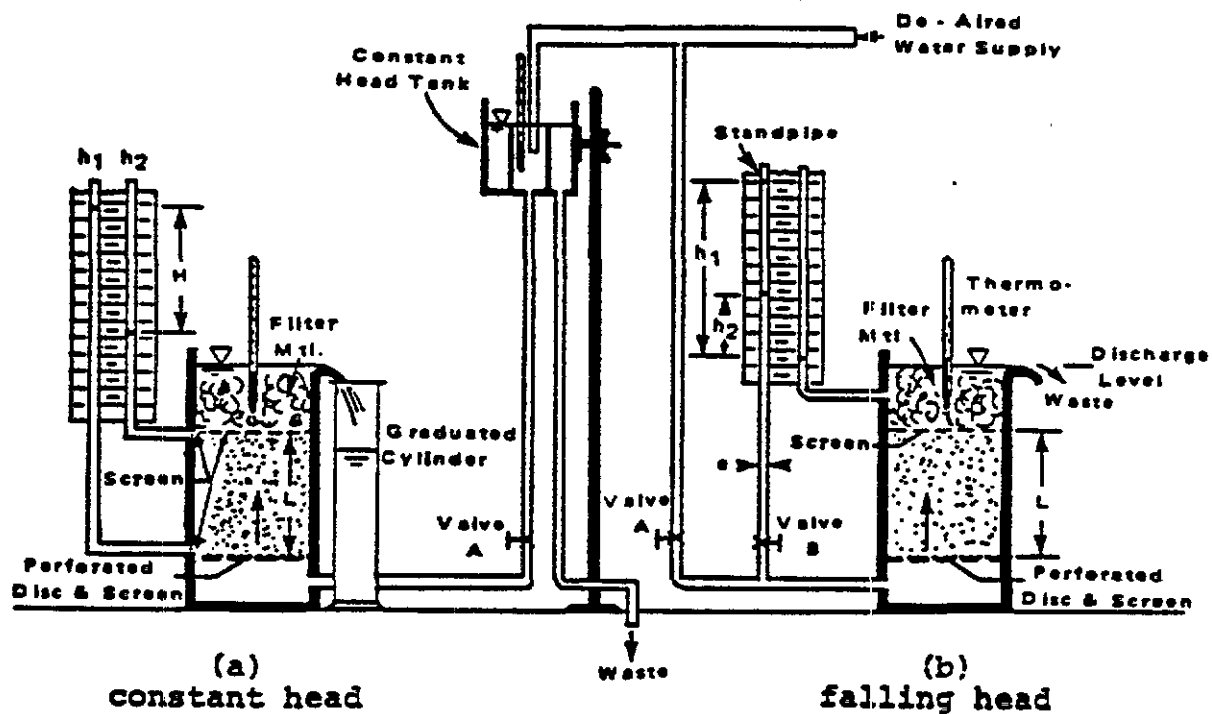


Figure 3.-- Apparatus setup for the constant head (a) and falling head (b) methods.

4. Mix the material thoroughly and place in the permeameter to avoid segregation. The material should be dropped just at the water surface, keeping the water surface about 1/2 in. above the top of the soil during placement. A funnel or a spoon is convenient for this purpose.
5. The placement procedure outlined above will result in a saturated specimen of uniform density although in a relatively loose condition. To produce a higher density in the specimen, the sides of the permeameter containing the soil sample are tapped uniformly along its circumference and length with a rubber mallet to produce an increase in density; however, extreme caution should be exercised so that fines are not put into suspension and segregated within the sample. As an alternative to this procedure, the specimen may be placed using an appropriate sized funnel or spoon. Compacting the specimen in layers is not recommended, as a film of dust which might affect the permeability results may be formed at the surface of the compacted layer. After placement, apply a vacuum to the top of the specimen and permit water to enter the evacuated specimen through the base of the permeameter.
6. After the specimen has been placed, weigh the excess material, if any, and the container. The specimen weight is the difference between the original weight of sample and the weight of the excess material. Care must be taken so that no material is lost during placement of the specimen. If there is evidence that material has been lost, oven-dry the specimen and weigh after the test as a check.
7. Level the top of the specimen, cover with a wire screen similar to that used at the base, and fill the remainder of the permeameter with a filter material.
8. Measure the length of the specimen, inside diameter of the permeameter, and distance between the centers of the manometer tubes (L) where they enter the permeameter.

2.5.4 Test procedure:

1. Adjust the height of the constant-head tank to obtain the desired hydraulic gradient. The hydraulic gradient should be selected so that the flow through the specimen is laminar. Hydraulic gradients ranging from 0.2 to 0.5 are recommended. Too high a hydraulic gradient may cause turbulent flow and also result in piping of soils. In general, coarser soils require lower hydraulic gradients. See Section 1.5 for further discussion of excessive gradients.
2. Open valve A (see Figure 3a) and record the initial piezometer readings after the flow has become stable. Exercise care in building up heads in the permeameter so that the specimen is not disturbed.

3. After allowing a few minutes for equilibrium conditions to be reached, measure by means of a graduated cylinder the quantity of discharge corresponding to a given time interval. Measure the piezometric heads (h_1 and h_2) and the water temperature in the permeameter.
4. Record the quantity of flow, piezometer readings, water temperature, and the time interval during which the quantity of flow was measured.

2.5.5 Calculations: By plotting the accumulated quantity of outflow versus time on rectangular coordinate paper, the slope of the linear portion of the curve can be determined, and the hydraulic conductivity can be calculated using Equation (8). The value of h in Equation (8) is the difference between h_1 and h_2 .

2.6 Falling-head test with conventional permeameter:

2.6.1 Applicability: The falling-head test can be used for all soil types, but is usually most widely applicable to materials having low permeability. Compacted, remolded, fine-grained soils can be tested with this method. This method presented is taken from the Engineering and Design, Laboratory Soils Testing Manual (U.S. Army, 1980).

2.6.2 Apparatus: The schematic diagram of the falling-head permeameter is shown in Figure 3b. The permeameter consists of the following equipment:

1. Permeameter cylinder, a transparent acrylic cylinder having a diameter at least 8 times the diameter of the largest particles;
2. Porous disk;
3. Wire screen;
4. Filter materials;
5. Manometer;
6. Timing device; and

2.6.3 Sample Preparation: Sample preparation for coarse-grained soils is similar to that described previously in Section 2.4.3. For fine-grained soils, samples are compacted to the desired density using methods described in ASTM Method D698-70.

2.6.4 Test Procedure:

1. Measure and record the height of the specimen, L , and the cross-sectional area of the specimen, A .

2. With valve B open (see Figure 3b), crack valve A, and slowly bring the water level up to the discharge level of the permeameter.
3. Raise the head of water in the standpipe above the discharge level of the permeameter. The difference in head should not result in an excessively high hydraulic gradient during the test. Close valves A and B.
4. Begin the test by opening valve B. Start the timer. As the water flows through the specimen, measure and record the height of water in the standpipe above the discharge level, h_1 , at time t_1 , and the height of water above the discharge level, h_2 at time t_2 .

2.6.5 Calculations. From the test data, plot the logarithm of head versus time on rectangular coordinate paper, or use semi-log paper. The slope of the linear part of the curve is used to determine $\log_{10}(h_1/h_2)/t$. Calculate the hydraulic conductivity using Equation (9).

2.7 Modified compaction permeameter method:

2.7.1 Applicability: This method can be used to determine the hydraulic conductivity of a wide range of materials. The method is generally used for remolded fine-grained soils. The method is generally used under constant-head conditions. The method was taken from Anderson and Brown, 1981, and EPA (1980). It should be noted that this method method of Section 2.9.

2.7.2 Apparatus: The apparatus is shown in Figure 4 and consists of equipment and accessories as follows:

1. Soil chamber, a compaction mold having a diameter 8 times larger than the diameter of the largest particles (typically, ASTM standard mold, Number CN405, is used);
2. Fluid chamber, a compaction mold sleeve having the same diameter as the soil chamber;
3. 2-kg hammer;
4. Rubber rings used for sealing purposes;
5. A coarse porous stone having higher permeability than the tested sample;
6. Regulated source of compressed air; and
7. Pressure gage or manometer to determine the pressure on the fluid chamber.

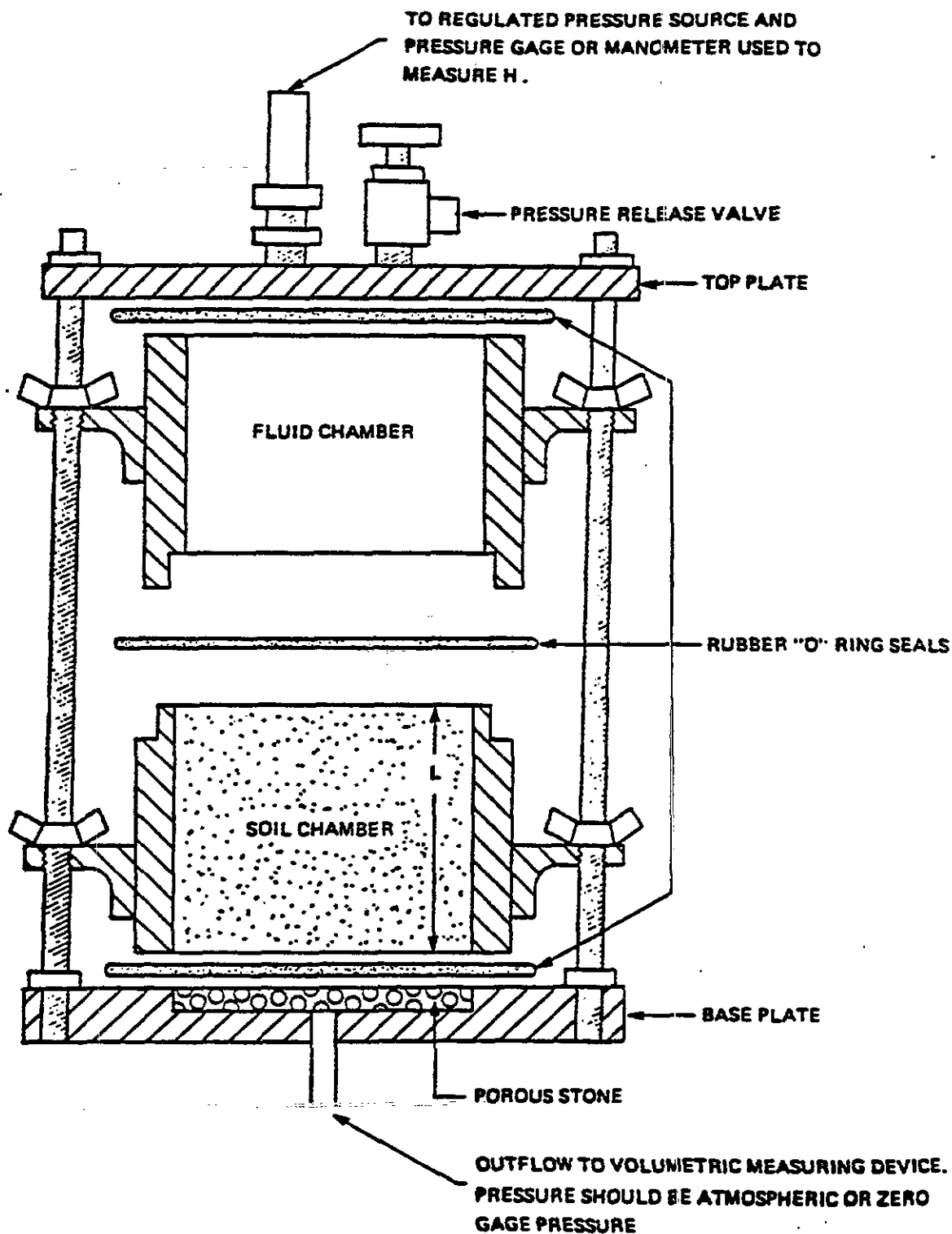


Figure 4.--Modified compaction permeameter.

Note: h in Equation B is the difference between the regulated inflow pressure and the outflow pressure. Source: Anderson and Brown, 1981.

2.7.3 Sample preparation:

1. Obtain sufficient representative soil sample. Air dry the sample at room temperature. Do not oven dry.
2. Thoroughly mix the selected representative sample with water to obtain a desired moisture content.
3. Compact the sample to the desired density within the mold using the method described as part of ASTM Method D698-70.
4. Level the surface of the compacted sample with straight edge, weigh and determine the density of the sample.
5. Measure the length and diameter of the sample.
6. Assemble the apparatus, make sure that there are no leaks, and then connect the pressure line to the apparatus.

2.7.4 Test procedure:

1. Place sufficient volume of water in the fluid chamber above the soil chamber.
2. Apply air pressure gradually to flush water through the sample until no air bubbles in the outflow are observed. For fine-grained soils, the saturation may take several hours to several days, depending on the applied pressure.
3. After the sample is saturated, measure and record the quantity of outflow versus time.
4. Record the pressure reading (h) on the top of the fluid chamber when each reading is made.
5. Plot the accumulated quantity of outflow versus time on rectangular coordinate paper.
6. Stop taking readings as soon as the linear position of the curve is defined.

2.7.5 Calculations: The hydraulic conductivity can be calculated using Equation (8).

2.8 Triaxial-cell method with back pressure:

2.8.1 Applicability: This method is applicable for all soil types, but especially for fine-grained, compacted, cohesive soils in which full fluid saturation of the sample is difficult to achieve. Normally, the test is run under constant-head conditions.

2.8.2 Apparatus: The apparatus is similar to conventional triaxial apparatus. The schematic diagram of this apparatus is shown in Figure 5.

2.8.3 Sample preparation: Disturbed or undisturbed samples can be tested. Undisturbed samples must be trimmed to the diameter of the top cap and base of the triaxial cell. Disturbed samples should be prepared in the mold using either kneading compaction for fine-grained soils, or by the pouring and vibrating method for coarse-grained soils, as discussed in Section 2.5.3.

2.8.4 Test procedure:

1. Measure the dimensions and weight of the prepared sample.
2. Place one of the prepared specimens on the base.
3. Place a rubber membrane in a membrane stretcher, turn both ends of the membrane over the ends of the stretcher, and apply a vacuum to the stretcher. Carefully lower the stretcher and membrane over the specimen. Place the specimen and release the vacuum on the membrane stretcher. Turn the ends of the membrane down around the base and up around the specimen cap and fasten the ends with O-rings.
4. Assemble the triaxial chamber and place it in position in the loading device. Connect the tube from the pressure reservoir to the base of the triaxial chamber. With valve C (see Figure 5) on the pressure reservoir closed and valves A and B open, increase the pressure inside the reservoir, and allow the pressure fluid to fill the triaxial chamber. Allow a few drops of the pressure fluid to escape through the vent valve (valve B) to insure complete filling of the chamber with fluid. Close valve A and the vent valve.
5. Place saturated filter paper disks having the same diameter as that of the specimen between the specimen and the base and cap; these disks will also facilitate removal of the specimen after the test. The drainage lines and the porous inserts should be completely saturated with deaired water. The drainage lines should be as short as possible and made of thick-walled, small-bore tubing to insure minimum elastic changes in volume due to changes in pressure. Valves in the drainage lines (valves E, F, and G in Figure 5) should preferably be of a type which will cause no discernible change of internal volume when operated. While mounting the specimen in the compression chamber, care should be exercised to avoid entrapping any air beneath the membrane or between the specimen and the base and cap.

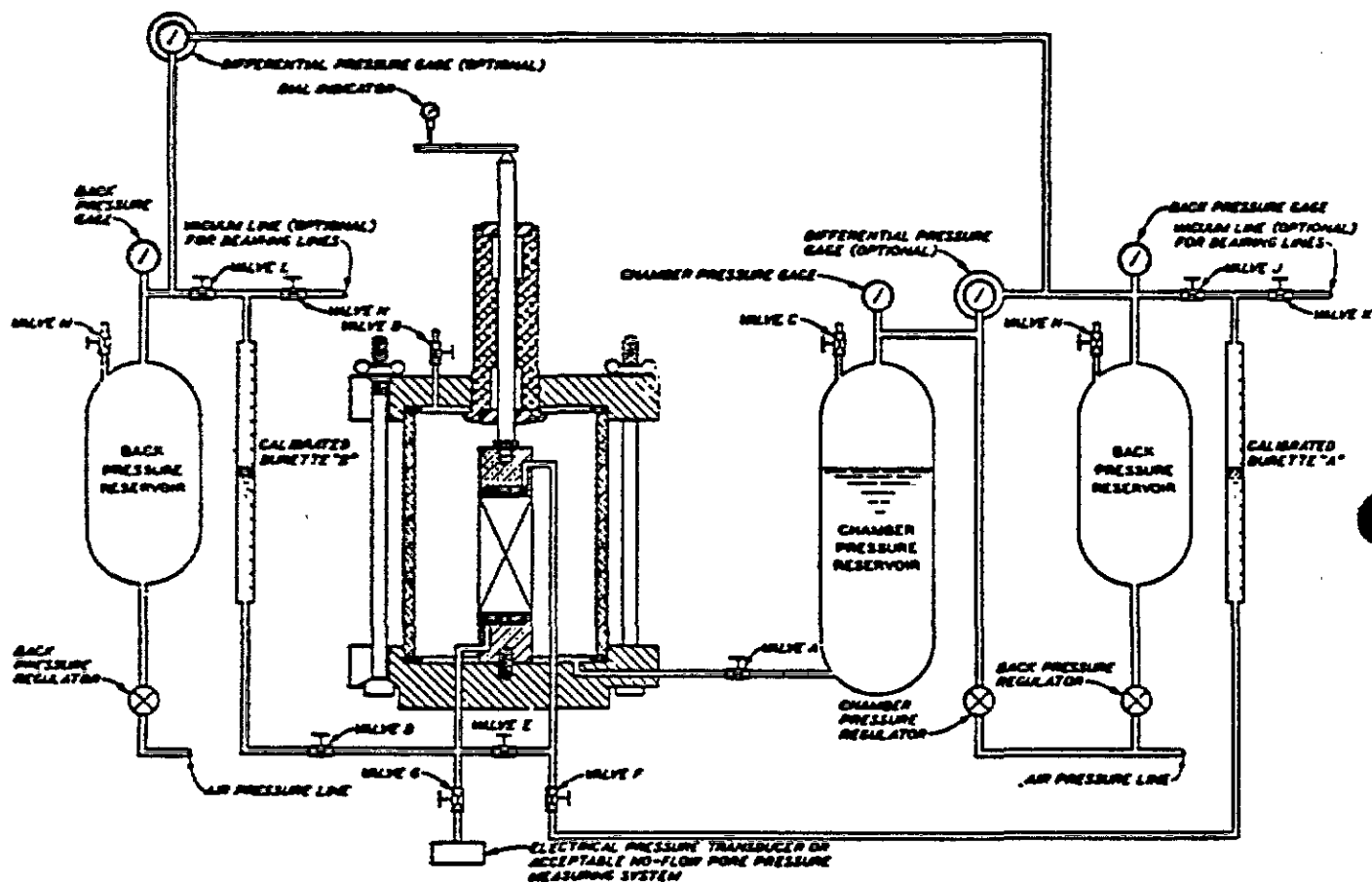


Figure 5.--Schematic diagram of typical triaxial compression apparatus for hydraulic conductivity tests with back pressure.

Source: U.S. Army Corps of Engineers, 1970

6. For ease and uniformity of saturation, as well as to allow volume changes during consolidation to be measured with the burette, specimens should be completely saturated before any appreciable consolidation is permitted; therefore, the difference between the chamber pressure and the back pressure should not exceed 5 psi during the saturation phase. To insure that a specimen is not prestressed during the saturation phase, the back pressure must be applied in small increments, with adequate time between increments to permit equalization of pore water pressure throughout the specimen.
7. With all valves closed, adjust the pressure regulators to a chamber pressure of about 7 psi and a back pressure of about 2 psi. Now open valve A to apply the preset pressure to the chamber fluid and simultaneously open valve F to apply the back pressure through the specimen cap. Immediately open valve G and read and record the pore pressure at the specimen base. When the measured pore pressure becomes essentially constant, close valves F and G and record the burette reading.
8. Using the technique described in Step 3, increase the chamber pressure and the back pressure in increments, maintaining the back pressure at about 5 psi less than the chamber pressure. The size of each increment might be 5, 10, or even 20 psi, depending on the compressibility of the soil specimen and the magnitude of the desired consolidation pressure. Open valve G and measure the pore pressure at the base immediately upon application of each increment of back pressure and observe the pore pressure until it becomes essentially constant. The time required for stabilization of the pore pressure may range from a few minutes to several hours depending on the permeability of the soil. Continue adding increments of chamber pressure and back pressure until, under any increment, the pore pressure reading equals the applied back pressure immediately upon opening valve G.
9. Verify the completeness of saturation by closing valve F and increasing the chamber pressure by about 5 psi. The specimen shall not be considered completely saturated unless the increase in pore pressure immediately equals the increase in chamber pressure.
10. When the specimen is completely saturated, increase the chamber pressure with the drainage valves closed to attain the desired effective consolidation pressure (chamber pressure minus back pressure). At zero elapsed time, open valves E and F.
11. Record time, dial indicator reading, and burette reading at elapsed times of 0, 15, and 30 sec, 1, 2, 4, 8, and 15 min, and 1, 2, 4, and 8 hr, etc. Plot the dial indicator readings and

burette readings on an arithmetic scale versus elapsed time on a log scale. When the consolidation curves indicate that primary consolidation is complete, close valves E and F.

12. Apply a pressure to burette B greater than that in burette A. The difference between the pressures in burettes B and A is equal to the head loss (h); h divided by the height of the specimen after consolidation (L) is the hydraulic gradient. The difference between the two pressures should be kept as small as practicable, consistent with the requirement that the rate of flow be large enough to make accurate measurements of the quantity of flow within a reasonable period of time. Because the difference in the two pressures may be very small in comparison to the pressures at the ends of the specimen, and because the head loss must be maintained constant throughout the test, the difference between the pressures within the burettes must be measured accurately; a differential pressure gage is very useful for this purpose. The difference between the elevations of the water within the burettes should also be considered (1 in. of water = 0.036 psi of pressure).
13. Open valves D and F. Record the burette readings at any zero elapsed time. Make readings of burettes A and B and of temperature at various elapsed times (the interval between successive readings depends upon the permeability of the soil and the dimensions of the specimen). Plot arithmetically the change in readings of both burettes versus time. Continue making readings until the two curves become parallel and straight over a sufficient length of time to determine accurately the rate of flow as indicated by the slope of the curves.

2.8.5 Calculations: The hydraulic conductivity can be calculated using Equation (8).

2.9 Pressure-chamber permeameter method:

2.9.1 Applicability: This method can be used to determine hydraulic conductivity of a wide range of soils. Undisturbed and disturbed samples can be tested under falling-head conditions using this method. This method is also applicable to both coarse- and fine-grained soils, including remolded, fine-grained materials.

2.9.2 Apparatus: The apparatus, shown in Figure 6, consists of

1. Pressure chamber;
2. Standpipe;
3. Specimen cap and base; and
4. Coarse porous plates.

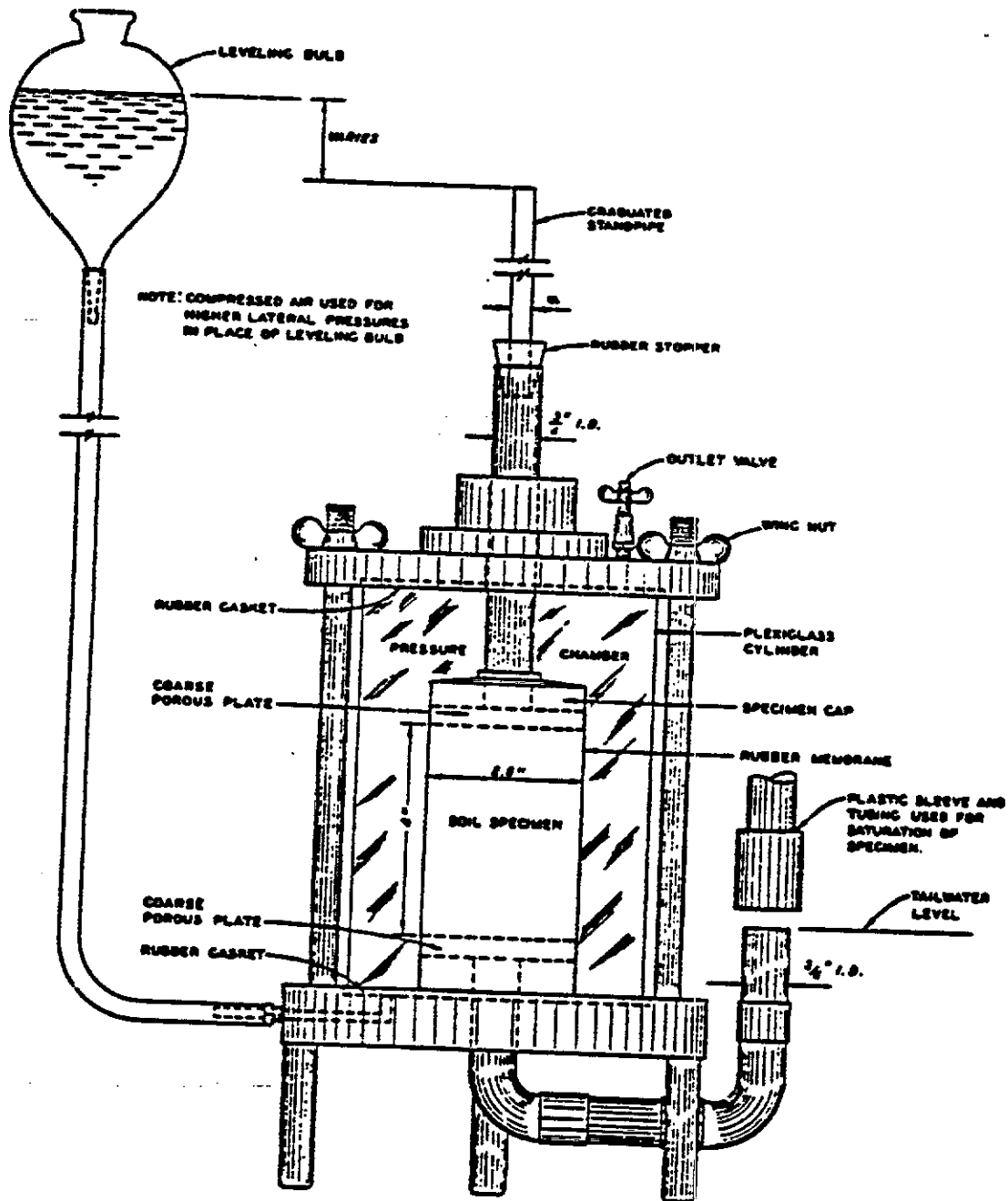


Figure 6.--Pressure chamber for hydraulic conductivity.
Source: U.S. Army Corps of Engineers, 1980.

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The apparatus is capable of applying confining pressure to simulate field stress conditions.

2.9.3 Sample preparation: The sample preparation of disturbed and undisturbed conditions can be prepared in the chamber and enclosed within the rubber membrane, as discussed in Section 2.8.4.

2.9.4 Test procedure:

1. By adjusting the leveling bulb, a confining pressure is applied to the sample such that the stress conditions represent field conditions. For higher confining pressure, compressed air may be used.
2. Allow the sample to consolidate under the applied stress until the end of primary consolidation.
3. Flush water through the sample until no indication of air bubbles is observed. For higher head of water, compressed air may be used.
4. Adjust the head of water to attain a desired hydraulic gradient.
5. Measure and record the head drop in the standpipe along with elapsed time until the plot of logarithm of head versus time is linear for more than three consecutive readings.

2.9.5 Calculations: The hydraulic conductivity can be determined using Equation (9).

2.10 Sources of error for laboratory test for hydraulic conductivity: There are numerous potential sources of error in laboratory tests for hydraulic conductivity. Fixed-wall permeameters may have problems with sidewall leakage, causing higher values of hydraulic conductivity. Flexible-membrane permeameters may yield misleadingly low values for hydraulic conductivity when testing with a leachate that causes contraction and shrinkage cracks in the sample because the membrane shrinks with the sample. Table B summarizes some potential errors that can occur. Olsen and Daniel (1981) provide a more detailed explanation of sources of these errors and methods to minimize them. If the hydraulic conductivity does not fall within the expected range for the soil type, as given in Table C, the measurement should be repeated after checking the source of error in Table B.

TABLE B

SUMMARY OF PUBLISHED DATA ON POTENTIAL ERRORS
IN USING DATA FROM
LABORATORY PERMEABILITY TESTS ON SATURATED SOILS

Source of Error (References)	Measured K	Too Low or Too High?
1. Voids formed in sample preparation (Olsen and Daniel, 1981).		High
2. Smear zone formed during trimming (Olsen and Daniel, 1981).		Low
3. Use of distilled water as a permeant (Fireman, 1944; and Wilkinson, 1969).		Low
4. Air in sample (Johnson, 1954)		Low
5. Growth of micro-organisms (Allison, 1947).		Low
6. Use of excessive hydraulic gradient (Schwartzendruber, 1968; and Mitchell and Younger, 1967).		Low or High
7. Use of temperature other than the test temperature.		Varies
8. Ignoring volume change due to stress change, with no confining pressure used.		High
9. Performing laboratory rather than in-situ tests (Olsen and Daniel, 1981).		Usually Low
10. Impedance caused by the test apparatus, including the resistance of the screen or porous stone used to support the sample.		Low

TABLE C

HYDRAULIC CONDUCTIVITIES ESTIMATED FROM GRAIN-SIZE DESCRIPTIONS
(In Feet Per Day)

Grain-Size Class or Range From Sample Description	Degree of Sorting			Silt Content		
	Poor	Moderate	Well	Slight	Moderate	High
<u>Fine-Grained Materials</u>						
Clay				Less than .001		
Silt, clayey				1 - 4		
Silt, slightly sandy				5		
Silt, moderately sandy				7 - 8		
Silt, very sandy				9 - 11		
Sandy silt				11		
Silty sand				13		
<u>Sands and gravels⁽¹⁾</u>						
Very fine sand	13	20	27	23	19	13
Very fine to fine sand	27	27	-	24	20	13
Very fine to medium sand	36	41-47	-	32	27	21
Very fine to coarse sand	48	-	-	40	31	24
Very fine to very coarse sand	59	-	-	51	40	29
Very fine sand to fine gravel	76	-	-	67	52	38
Very fine sand to medium gravel	99	-	-	80	66	49
Very fine sand to coarse gravel	128	-	-	107	86	64
Fine sand	27	40	53	33	27	20
Fine to medium sand	53	67	-	48	39	30
Fine to coarse sand	57	65-72	-	53	43	32
Fine to very coarse sand	70	-	-	60	47	35
Fine sand to fine gravel	88	-	-	74	59	44
Fine sand to medium gravel	114	-	-	94	75	57
Fine sand to coarse gravel	145	-	-	107	87	72
Medium sand	67	80	94	64	51	40
Medium to coarse sand	74	94	-	72	57	42
Medium to very coarse sand	84	98-111	-	71	61	49
Medium sand to fine gravel	103	-	-	84	68	52
Medium sand to medium gravel	131	-	-	114	82	66
Medium sand to coarse gravel	164	-	-	134	108	82
Coarse sand	80	107	134	94	74	53
Coarse to very coarse sand	94	134	-	94	75	57
Coarse sand to fine gravel	116	136-156	-	107	88	68
Coarse sand to medium gravel	147	-	-	114	94	74
Coarse sand to coarse gravel	184	-	-	134	100	92

(1) Reduce by 10 percent if grains are subangular.
Source: Lappala (1978).

(continued)

TABLE C (Continued)

Grain-Size Class or Range From Sample Description	Degree of Sorting			Silt Content		
	Poor	Moderate	Well	Slight	Moderate	High
<u>Sands and Gravels</u> (1)						
Very coarse sand	107	147	187	114	94	74
Very coarse sand to fine gravel	134	214	-	120	104	87
Very coarse sand to medium gravel	1270	199-227	-	147	123	99
Very coarse sand to coarse gravel	207	-	-	160	132	104
Fine gravel	160	214	267	227	140	107
Fine to medium gravel	201	334	-	201	167	134
Fine to coarse gravel	245	289-334	-	234	189	144
Medium gravel	241	231	401	241	201	160
Medium to coarse gravel	294	468	-	294	243	191
Coarse gravel	334	468	602	334	284	234

(1) Reduce by 10 percent if grains are subangular.
Source: Lappala (1978).

2.11 Leachate conductivity using laboratory methods: Many primary and secondary leachates found at disposal sites may be nonaqueous liquids or aqueous fluids of high ionic strength. These fluids may significantly alter the intrinsic permeability of the porous medium. For example, Anderson and Brown (1981) have demonstrated increases in hydraulic conductivity of compacted clays of as much as two orders of magnitude after the passage of a few pore volumes of a wide range of organic liquids. Consequently, the effects of leachate on these materials should be evaluated by laboratory testing. The preceding laboratory methods can all be used to determine leachate conductivity by using the following guidelines.

2.11.1 Applicability: The determination of leachate conductivity may be required for both fine-grained and coarse-grained materials. Leachates may either increase or decrease the hydraulic conductivity. Increases are of concern for compacted clay liners, and decreases are of concern for drain materials. The applicability sections of the preceding methods should be used for selecting an appropriate test for leachate conductivity. The use of the modified compaction method (Section 2.7) for determining leachate conductivity is discussed extensively in EPA Publication SW870 (EPA 1980).

2.11.2 Leachate used: A supply of leachate must be obtained that is as close in chemical and physical properties to the anticipated leachate at the disposal site as possible. Methods for obtaining such leachate are beyond the scope of this report. However, recent publications by EPA (1979) and Conway and Malloy (1981) give methodologies for simulating the leaching environment to obtain such leachate. Procedures for deairing the leachate supply are given in Section 2.4. The importance of preventing bacterial growth in leachate tests will depend on the expected conditions at the disposal site. The chemical and physical properties that may result in corrosion, dissolution, or encrustation of laboratory hydraulic conductivity apparatus should be determined prior to conducting a leachate conductivity test. Properties of particular importance are the pH and the vapor pressure of the leachate. Both extremely acidic and basic leachates may corrode materials. In general, apparatus for leachate conductivity tests should be constructed of inert materials, such as acrylic plastic, nylon, or Teflon. Metal parts that might come in contact with the leachate should be avoided. Leachates with high vapor pressures may require special treatment. Closed systems for fluid supply and pressure measurement, such as those in the modified triaxial-cell methods, should be used.

2.11.3 Safety: Tests involving the use of leachates should be conducted under a vented hood, and persons conducting the tests should wear appropriate protective clothing and eye protection. Standard laboratory safety procedures such as those as given by Manufacturing Chemists Association (1971) should be followed.

2.11.4 Procedures: The determination of leachate conductivity should be conducted immediately following the determination of hydraulic

conductivity (Anderson and Brown, 1981). This procedure maintains fluid saturation of the sample, and allows a comparison of the leachate and hydraulic conductivities under the same test conditions. This procedure requires modifications of test operations as described below.

2.11.5 Apparatus: In addition to a supply reservoir for water as shown in Figures 3 through 6, a supply reservoir for leachate is required. Changing the inflow to the test cell from water to leachate can be accomplished by providing a three-way valve in the inflow line that is connected to each of the reservoirs.

2.11.6 Measurements: Measurements of fluid potential and outflow rates are the same for leachate conductivity and hydraulic conductivity. If the leachate does not alter the intrinsic permeability of the sample, the criteria for the time required to take measurements is the same for leachate conductivity tests as for hydraulic conductivity tests. However, if significant changes occur in the sample by the passage of leachate, measurements should be taken until either the shape of a curve of conductivity versus pore volume can be defined, or until the leachate conductivity exceeds the applicable design value for hydraulic conductivity.

2.11.7 Calculations: If the leachate conductivity approaches a constant value, Equations (8) and (9) can be used. If the conductivity changes continuously because of the action of the leachate, the following modifications should be made. For constant-head tests, the conductivity should be determined by continuing a plot of outflow volume versus time for the constant rate part of the test conducted with water. For falling-head tests, the slope of the logarithm of head versus time should be continued.

2.11.7.1 If the slope of either curve continues to change after the flow of leachate begins, the leachate is altering the intrinsic permeability of the sample. The leachate conductivity in this case is not a constant. In this case, values of the slope of the outflow curve to use in Equation (8) or (9) must be taken as the tangent to the appropriate outflow curve at the times of measurement.

3.0 FIELD METHODS

This section discusses methods available for the determination of fluid conductivity under field conditions. As most of these tests will use water as the testing fluid, either natural formation water or water added to a borehole or piezometer, the term hydraulic conductivity will be used for the remainder of this section. However, if field tests are run with leachate or other fluids, the methods are equally applicable.

The location of wells, selection of screened intervals, and the appropriate tests that are to be conducted depend upon the specific site under

investigation. The person responsible for such selections should be a qualified hydrogeologist or geotechnical engineer who is experienced in the application of established principles of contaminant hydrogeology and ground water hydraulics. The following are given as general guidelines.

1. The bottom of the screened interval should be below the lowest expected water level.
2. Wells should be screened in the lithologic units that have the highest probability of either receiving contaminants or conveying them down gradient.
3. Wells up gradient and down gradient of sites should be screened in the same lithologic unit.

Standard reference texts on ground water hydraulics and contaminant hydrogeology that should be consulted include: Bear (1972), Bouwer (1978), Freeze and Cherry (1979), Stallman (1971), and Walton (1970).

The success of field methods in determining hydraulic conductivity is often determined by the design, construction, and development of the well or borehole used for the tests. Details of these methods are beyond the scope of this report; however, important considerations are given in Sections 3.1 and 3.2. Detailed discussions of well installation, construction, and development methods are given by Bouwer, pp. 160-180 (1978), Acker (1974), and Johnson (1972).

The methods for field determination of hydraulic conductivity are restricted to well or piezometer type tests applicable below existing water tables. Determinations of travel times of leachate and dissolved solutes above the water table usually require the application of unsaturated flow theory and methods which are beyond the scope of this report.

3.1 Well-construction considerations: The purpose of using properly constructed wells for hydraulic conductivity testing is to assure that test results reflect conditions in the materials being tested, rather than conditions caused by well construction. In all cases, diagrams showing all details of the actual well or borehole constructed for the test should be made. Chapter 3 of the U.S. EPA, RCRA Ground Water Monitoring Technical Enforcement Guidance Document (TEGD) should be consulted.

3.1.1 Well installation methods: Well installation methods are listed below in order of preference for ground water testing and monitoring. The order was determined by the need to minimize side-wall plugging by drilling fluids and to maximize the accurate detection of saturated zones. This order should be used as a guide, combined with the judgment of an experienced hydrogeologist in selecting a drilling method. The combined uses of wells for hydraulic conductivity testing, water-level monitoring, and water-quality sampling for organic contaminants were considered in arriving at the ranking.

1. Hollow-stem auger;
2. Cable tool;
3. Air rotary;
4. Rotary drilling with non-organic drilling fluids;
5. Air foam rotary; and
6. Rotary with organic-based drilling fluids.

Although the hollow stem-auger method is usually preferred for the installation of most shallow wells (less than 100 feet), care must be taken if the tested zone is very fine. Smearing of the borehole walls by drilling action can effectively seal off the borehole from the adjacent formation. Scarification can be used to remedy this.

3.1.2 Wells requiring well screens: Well screens placed opposite the interval to be tested should be constructed of materials that are compatible with the fluids to be encountered. Generally an inert plastic such as PVC is preferred for ground water contamination studies. The screen slot size should be determined to minimize the inflow of fine-grained material to the well during development and testing. Bouwer (1978) and Johnson (1972) give a summary of guidelines for sizing well screens.

3.1.2.1 The annulus between the well screen and the borehole should be filled with an artificial gravel pack or sand filter. Guidelines for sizing these materials are given by Johnson (1972). For very coarse materials, it may be acceptable to allow the materials from the tested zone to collapse around the screen forming a natural gravel pack.

3.1.2.2 The screened interval should be isolated from overlying and underlying zones by materials of low hydraulic conductivity. Generally, a short bentonite plug is placed on top of the material surrounding the screen, and cement grout is placed in the borehole to the next higher screened interval (in the case of multiple screen wells), or to the land surface for single screen wells.

3.1.2.3 Although considerations for sampling may dictate minimum casing and screen diameters, the recommended guideline is that wells to be tested by pumping, bailing, or injection in coarse-grained materials should be at least 4-inches inside diameter. Wells to be used for testing materials of low hydraulic conductivity by sudden removal or injection of a known volume of fluid should be constructed with as small a casing diameter as possible to maximize measurement resolution of fluid level changes. Casing sizes of 1.25 to 1.50 inches usually allow this resolution while enabling the efficient sudden withdrawal of water for these tests.

3.1.3 Wells not requiring well screens: If the zone to be tested is sufficiently indurated that a well screen and casing are not required to prevent caving in, it is preferable to use a borehole open to the zone to be tested. These materials generally are those having low to extremely low hydraulic conductivities. Consolidated rocks having high conductivity because of the presence of fractures and solution openings may also be completed without the use of a screen and gravel pack. Uncased wells may penetrate several zones for which hydraulic conductivity tests are to be run. In these cases, the zones of interest can be isolated by the use of inflatable packers.

3.2 Well development: For wells that are constructed with well screens and gravel packs, and for all wells in which drilling fluids have been used that may have penetrated the materials to be tested, adequate development of the well is required to remove these fluids and to remove the fine-grained materials from the zone around the well screen. Development is carried out by methods such as intermittent pumping, jetting with water, surging, and bailing. Adequate development is required to assure maximum communication between fluids in the borehole and the zone to be tested. Results from tests run in wells that are inadequately developed will include an error caused by loss of fluid potential across the undeveloped zone, and computed hydraulic conductivities will be lower than the actual value. Bouwer (1978) and Johnson (1975) give further details on well development including methods to determine when adequate development has occurred. The U.S. EPA TEGD should also be consulted.

3.3 Data interpretation and test selection considerations: Hydraulic conductivity may be determined in wells that are either cased or uncased as described in Section 3.1. The tests all involve disturbing the existing fluid potential in the tested zone by withdrawal from or injection of fluid into a well, either as a slug over an extremely short period of time, or by continuous withdrawal or injection of fluid. The hydraulic conductivity is determined by measuring the response of the water level or pressure in the well as a function of time since the start of the test. Many excellent references are available that give the derivation and use of the methods that are outlined below, including Bouwer (1978), Walton (1969), and Lohman (1972).

3.3.1 The selection of a particular test method and data analysis technique requires the consideration of the purposes of the test, and the geologic framework in which the test is to be run. Knowledge of the stratigraphic relationships of the zone to be tested and both overlying and underlying materials should always be used to select appropriate test design and data interpretation methods.

3.3.2 The equations given for all computational methods given here and in the above references are based on idealized models comprising layers of materials of different hydraulic conductivities. The water-level response caused by disturbing the system by the addition or removal of water can be similar for quite different systems. For example, the response of a water-table aquifer and a leaky, confined aquifer to

pumping can be very similar. Consequently, it is not considered acceptable practice to obtain data from a hydraulic conductivity test and interpret the type of hydraulic system present without supporting geologic evidence.

3.3.3 The primary use of hydraulic conductivity data from tests described subsequently will usually be to aid in siting monitoring wells for facility design as well as for compliance with Subpart F of Part 264. As such, the methods are abbreviated to provide guidance in determining hydraulic conductivity only. Additional analyses that may be possible with some methods to define the storage properties of the aquifer are not included. The U.S. EPA TEGD has an expanded discussion on the relationship between K tests and siting design (Chapter 1) and should be consulted.

3.3.4 The well test methods are discussed under the following two categories: 1) methods applicable to coarse-grained materials and tight to extremely tight materials under confined conditions; and 2) methods applicable to unconfined materials of moderate permeability. The single well tests integrate the effects of heterogeneity and anisotropy. The effects of boundaries such as streams or less permeable materials usually are not detectable with these methods because of the small portion of the geologic unit that is tested.

3.4 Single well tests: The tests for determining hydraulic conductivity with a single well are discussed below based on methods for confined and unconfined conditions. The methods are usually called slug tests because the test involves removing a slug of water instantaneously from a well and measuring the recovery of water in the well. The method was first developed by Hvorslev (1951), whose analysis did not consider the effect of fluid stored in the well. Cooper and others (1967) developed a method that considers well bore storage. However, their method only applied to wells that are open to the entire zone to be tested and that tap confined aquifers. Because of the rapid water-level response in coarse materials, the tests are generally limited to zones with a transmissivity of less than about 70 cm²/sec (Lohman, 1972). The method has been extended to allow testing of extremely tight formations by Bredehoeft and Papadopoulos (1980). Bouwer and Rice (1976) developed a method for analyzing slug tests for unconfined aquifers.

3.4.1 Method for moderately permeable formations under confined conditions:

3.4.1.1 Applicability: This method is applicable for testing zones to which the entire zone is open to the well screen or open borehole. The method usually is used in materials of moderate hydraulic conductivity which allow measurement of water-level response over a period of a hour to a few days. More permeable zones can be tested with rapid response water-level recording equipment. The method assumes that the tested zone is uniform in all radial directions from the test well. Figure 7 illustrates the test geometry for this method.

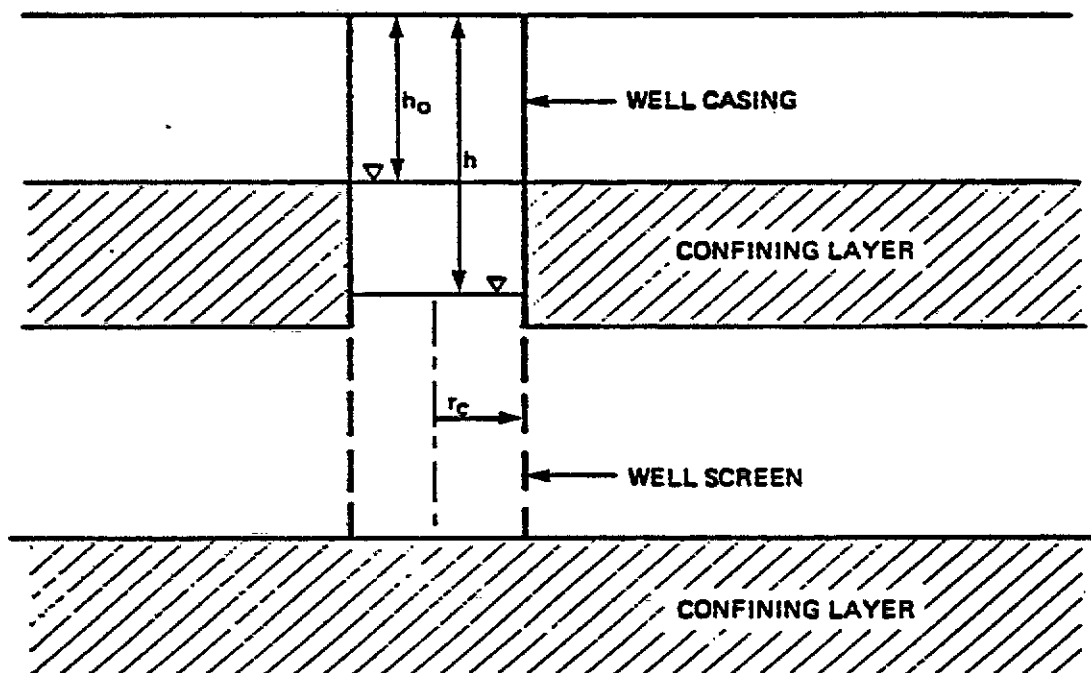


Figure 7.--Geometry and variable definition for slug tests in confined aquifers.

3.4.1.2 Procedures: The slug test is run by utilizing some method of removing or adding a known volume of water from the well bore in a very short time period and measuring the recovery of the water level in the well. The procedures are the same for both unconfined and confined aquifers. Water is most effectively removed by using a bailer that has been allowed to fill and stand in the well for a sufficiently long period of time so that any water-level disturbance caused by the insertion of the bailer will have reached equilibrium. In permeable materials, this recovery time may be as little as a few minutes. An alternate method of effecting a sudden change in water level is the withdrawal of a weighted float. The volume of water displaced can be computed using the known submersed volume of the float and Archimedes' principle (Lohman, 1972).

Water-level changes are recorded using either a pressure transducer and a strip chart recorder, a weighted steel tape, or an electric water-level probe. For testing permeable materials that approach or exceed 70 cm²/sec, a rapid-response transducer/recorder system is usually used because essentially full recovery may occur in a few minutes. Because the rate of water-level response decays with time, water-level or pressure changes should be taken at increments that are approximately equally spaced in the logarithm of the time since fluid withdrawal. The test should be continued until the water level in the well has recovered to at least 85 percent of the initial pre-test value.

3.4.1.3 Calculations: Calculations for determining hydraulic conductivity for moderately permeable formations under confined conditions can be made using the following procedure:

1. Determine the transmissivity of the tested zone by plotting the ratio h/h_0 on an arithmetic scale against time since removal of water (t) on a logarithmic scale. The observed fluid potential in the well during the test as measured by water level or pressure is h , and the fluid potential before the instant of fluid withdrawal is h_0 . The data plot is superimposed on type curves, such as those given by Lohman (1972), Plate 2, or plotted from Appendix A, with the h/h_0 and time axes coincident. The data plot is moved horizontally until the data fits one of the type curves. A value of time on the data plot corresponding to a dimensionless time (ρ) on the type curve plot is chosen, and the transmissivity is computed from the following:

$$T = \frac{\rho r_c^2}{t} \quad (10)$$

where:

r_c is the radius of the casing (Lohman, p. 29 (1972)).

The type curves plotted using data in Appendix A are not to be confused with those commonly referred to as "Theis Curves" which are used for pumping tests in confined aquifers (Lohman, 1972). The type curve method is a general technique of determining aquifer parameters when the solution to the descriptive flow equation involves more than one unknown parameter. Although both the storage coefficient and transmissivity of the tested interval can be determined with the type curve method for slug tests, determination of storage coefficients is beyond the scope of this report. See Section 3.4.1.4 for further discussion of the storage coefficient.

If the data in Appendix A are used, a type curve for each value of α is prepared by plotting $F(\alpha, \beta)$ on the arithmetic scale and dimensionless time (β) on the logarithmic scale of semi-log paper.

2. Determine the hydraulic conductivity by dividing the transmissivity (T) calculated above by the thickness of the tested zone.

3.4.1.4 Sources of error: The errors that can arise in conducting slug tests can be of three types: those resulting from the well or borehole construction; measurement errors; and data analysis error.

Well construction and development errors: This method assumes that the entire thickness of the zone of interest is open to the well screen or boreholes and that flow is principally radial. If this is not the case, the computed hydraulic conductivity may be too high. If the well is not properly developed, the computed conductivity will be too low.

Measurement errors: Determining or recording the fluid level in the borehole and the time of measurement incorrectly can cause measurement errors. Water levels should be measured to an accuracy of at least 1 percent of the initial water-level change. For moderately permeable materials, time should be measured with an accuracy of fractions of minutes, and, for more permeable materials, the time should be measured in terms of seconds or fractions of seconds. The latter may require the use of a rapid-response pressure transducer and recorder system.

Data analysis errors: The type curve procedure requires matching the data to one of a family of type curves, described by the parameter α , which is a measure of the storage in the well bore and aquifer. Papadopoulos and others (1973) show that an error of two orders of magnitude in the selection of α would result in an error of less than 30 percent in the value of transmissivity determined. Assuming no error in determining the thickness of the zone tested, this is equivalent to a 30 percent error in the hydraulic conductivity.

3.4.2 Methods for extremely tight formations under confined conditions:

3.4.2.1 Applicability: This test is applicable to materials that have low to extremely low permeability such as silts, clays, shales, and indurated lithologic units. The test has been used to determine hydraulic conductivities of shales of as low as 10^{-10} cm/sec.

3.4.2.2 Procedures: The test described by Bredehoeft and Papadopoulos (1980) and modified by Neuzil (1982) is conducted by suddenly pressurizing a packed-off zone in a portion of a borehole or well. The test is conducted using a system such as shown in Figure 8. The system is filled with water to a level assumed to be equal to the prevailing water level. (This step is required if sufficiently large times have not elapsed since the drilling of the well to allow full recovery of water levels.) A pressure transducer and recorder are used to monitor pressure changes in the system for a period prior to the test to obtain pressure trends preceding the test. The system is pressurized by addition of a known volume of water with a high-pressure pump. The valve is shut and the pressure decay is monitored. Neuzil's modification uses two packers with a pressure transducer below the bottom packer to measure the pressure change in the cavity and one between the two packers to monitor any pressure change caused by leakage around the bottom packer.

3.4.2.3 Calculations: The modified slug test as developed by Bredehoeft and Papadopoulos (1980) considered compressive storage of water in the borehole. These authors considered that the volume of the packed-off borehole did not change during the test and that all compressive storage resulted in compression of water under the pressure pulse. Neuzil (1980) demonstrated that under some test conditions this is not a valid assumption. The computation from either Lohman, Plate 2 (1972) or plotted from data given in Appendix A as described in Section 3.4.1.3. The values of time (t) and dimensionless time (β) are determined in the same manner as for the conventional tests. If compression of water only is considered, transmissivity is computed by replacing r_c by the quantity $(V_W C_W \rho g / \pi)$ in Equation 10:

$$T = \frac{\beta(V_W C_W \rho g / \pi)^2}{t} \quad (10)$$

where:

V_W is the volume of water in the packed-off cavity, L^3 ;

C_W is the compressibility of water, LT^2M^{-1} ;

ρ is the density of water, ML^{-3} ; and

g is the acceleration of gravity, LT^{-2} .

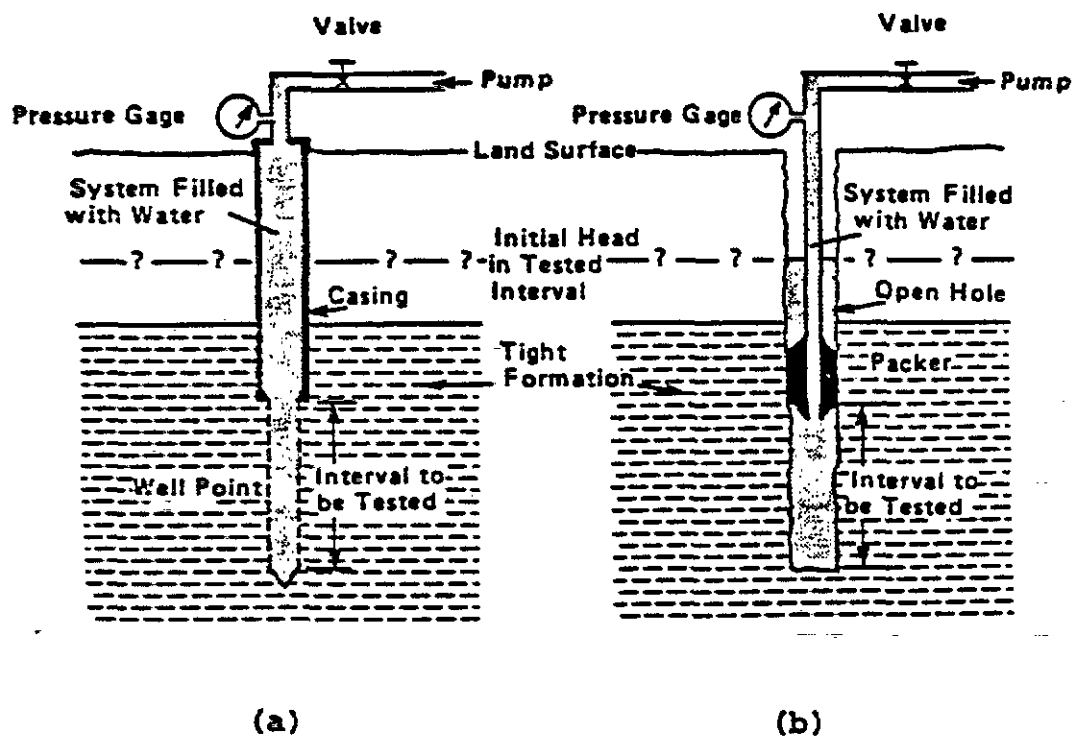


Figure 8.--Schematic diagram for pressurized slug test method in unconsolidated (a) and consolidated (b) materials. Source: Papadopoulos and Bredehoeft, 1980.

If the compressive storage is altered by changing the volume of the packed-off cavity (V), then the combined compressibility of the water and the expansion of the cavity (C_0) is used. C_0 is computed by measuring the volume of water injected during pressurization (ΔV) and the pressure change (ΔP) for the pressurization:

$$C_0 = \frac{\Delta V}{V \Delta P} \quad (11)$$

(Neuzil, p. 440 (1982)). Use of C_0 requires an accurate method of metering the volume of water injected and the volume of the cavity.

3.4.2.4 Sources of error: The types of errors in this method are the same as those for the conventional slug test. Errors may also arise by inaccurate determination of the cavity volume and volume of water injected. An additional assumption that is required for this method is that the hydraulic properties of the interval tested remain constant throughout the test. This assumption can best be satisfied by limiting the initial pressure change to a value only sufficiently large enough to be measured (Bredehoeft and Papadopoulos, 1980).

3.4.3 Methods for moderately permeable materials under unconfined conditions:

3.4.3.1 Applicability: This method is applicable to wells that fully or partially penetrate the interval of interest (Figure 9). The hydraulic conductivity determined will be principally the value in the horizontal direction (Bouwer and Rice, 1976).

3.4.3.2 Procedures: A general method for testing cased wells that partly or fully penetrate aquifers that have a water table as the upper boundary of the zone to be tested was developed by Bouwer and Rice (1976). The geometry and dimensions that are required to be known for the method are shown in Figure 9. The test is accomplished by effecting a sudden change in fluid potential in the well by withdrawal of either a bailer or submerged float as discussed in Section 3.4.1.2. Water-level changes can be monitored with either a pressure transducer and recorder, a wetted steel tape, or an electric water-level sounder. For highly permeable formations, a rapid-response transducer and recorder system is required. The resolution of the transducer should be about 0.01 m.

3.4.3.3 Calculations: The hydraulic conductivity is calculated using the following equation from Bouwer and Rice (1976), in the notation of this report:

$$K = \frac{r_c^2 \ln R/r_w}{2 L_e t} \ln \frac{y_0}{y} \quad (12)$$

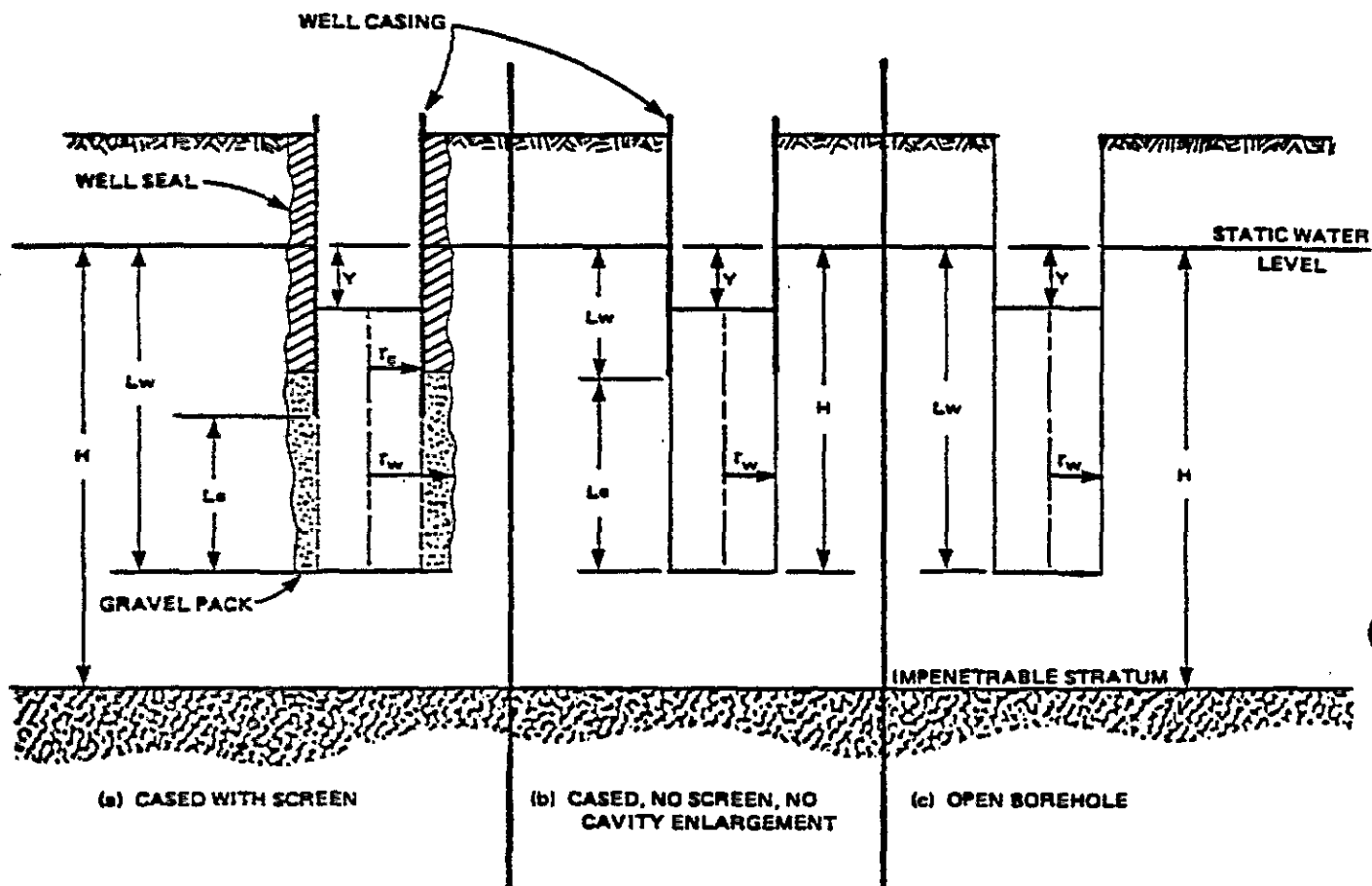


Figure 9.--Variable definitions for slug tests in unconfined materials. Cased wells are open at the bottom.

where r_c , r_w , L_e , t , Y , and K have been previously defined or are defined in Figure 8a. Y_0 is the value of Y immediately after withdrawal of the slug of water. The term R is an effective radius for wells that do not fully penetrate the aquifer that is computed using the following equation given by Bouwer and Rice (1976):

$$\ln \frac{R}{r_w} = \left\{ \frac{1.1}{\ln (L_w/r_w)} + \frac{A + B \ln [(H_0 - L_w)/r_w]}{(L_e/r_w)} \right\}^{-1} \quad (13)$$

If the quantity $(H_0 - L_w)/r_w$ is larger than 6, a value of 6 should be used.

For wells that completely penetrate the aquifer, the following equation is used:

$$\ln \frac{R}{r_w} = \left\{ \frac{1.1}{\ln (L_w/r_w)} + \frac{C}{L_e/r_w} \right\}^{-1} \quad (14)$$

(Bouwer, 1976). The values of the constants A , B , and C are given by Figure 10 (Bouwer and Rice, 1976).

For both cases, straight-line portions of plots of the logarithm of Y or Y_0/Y against time should be used to determine the slope, $(\ln Y_0/Y)/t$.

Additional methods for tests under unconfined conditions are summarized by Bower (1976) on pages 117-122. These methods are modifications of the cased-well method described above that apply either to an uncased borehole or to a well or piezometer in which the diameter of the casing and the borehole are the same (Figures 9b and 9c.)

3.4.3.4 Sources of error: The method assumes that flow of water from above is negligible. If this assumption cannot be met, the conductivities may be in error. Sufficient flow from the unsaturated zone by drainage would result in a high conductivity value. Errors caused by measuring water levels and recording time are similar to those discussed in Sections 3.4.1.4 and 3.4.2.4.

3.5 Multiple well tests: Hydraulic conductivity can also be determined by conventional pumping tests in which water is continuously withdrawn or injected using one well, and the water-level response is measured over time in or near more observation wells. The observation wells must be screened in the same strata as the injection or pumping well. These methods generally test larger portions of aquifers than the single well tests discussed in Section 3.4. For some circumstances these tests may be appropriate in obtaining data to use in satisfying requirements of Part 264 Subpart F. However, the large possibility for non-uniqueness in interpretation, problems involved in pumping contaminated fluids, and the expense of conducting such tests generally

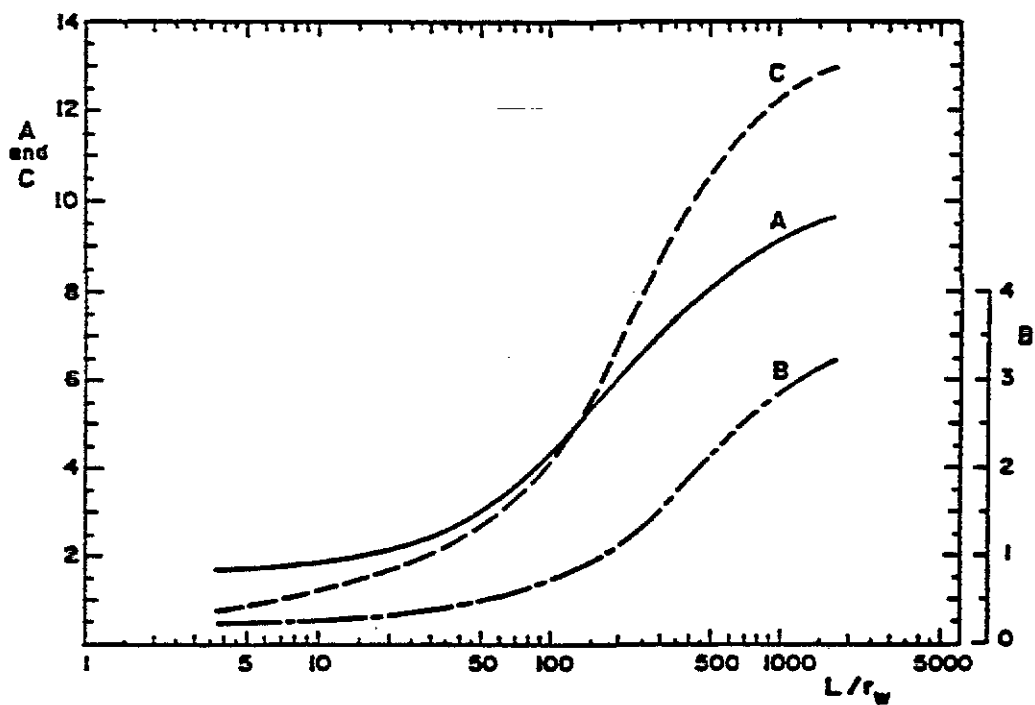


Figure 10. --Curves defining coefficients A, B, and C in equations 13 and 14 as a function of the ratio L/r_w .
Source: Bower and Rice, 1976.

preclude their use in problems of contaminant hydrogeology. The following references give excellent discussions of the design and interpretation of these tests: Lohman (1972), Stallman (1971), and Walton (1970).

3.6 Estimates of hydraulic conductivity for coarse-grained materials: The characterization of ground water flow systems to satisfy the intent of Part 264 Subpart F is preferably done with flow nets based on borehole measurements rather than relying on interpolation from grain-size analyses.

An empirical approach that has been used by the U.S. Geological Survey (Lappala, 1978) in several studies relates conductivity determined by aquifer testing to grain-size, degree of sorting and silt content. Table C provides the estimates of hydraulic conductivity.

Although estimates of K from analysis of grain-size and degree of sorting do provide a rough check on test values of K, repeated slug tests provide a better check on the accuracy of results.

3.7 Consolidation tests: As originally defined by Terzaghi (Terzaghi and Peck, 1967) the coefficient of consolidation (C_v) of a saturated, compressible, porous medium is related to the hydraulic conductivity by:

$$C_v = \frac{K}{\rho g \alpha} \quad (15)$$

where:

K is the hydraulic conductivity, LT⁻²;

ρ is the fluid density, ML⁻³;

g is the gravitational constant, LT⁻²; and

α is the soil's compressibility, LM⁻¹T².

The compressibility can be determined in the laboratory with several types of consolidometers, and is a function of the applied stress and the previous loading history. Lambe (1951) describes the testing procedure.

3.7.1 The transfer value of results from this testing procedure is influenced by the extent to which the laboratory loading simulates field conditions and by the consolidation rate. The laboratory loadings will probably be less than the stress that remolded clay liner will experience; therefore, the use of an already remolded sample in the consolidometer will probably produce no measurable results. This suggests that the test is of little utility in determining the hydraulic conductivity of remolded or compacted, fine-grained soils. Second, the consolidation rate determines the length of the testing period. For granular soils, this rate is fairly rapid. For fine-grained soils, the rate may be sufficiently slow that the previously described methods,

which give faster results, will be preferable. Cohesive soils (clays) must be trimmed from undisturbed samples to fit the mold, while cohesionless sands can be tested using disturbed, repacked samples (Freeze and Cherry, 1979).

3.7.2 In general, EPA believes that consolidation tests can provide useful information for some situations, but prefers the previously described methods because they are direct measurements of hydraulic conductivity. Hydraulic conductivity values determined using consolidation tests are not to be used in permit applications.

3.8 Fractured media: Determining the hydraulic properties of fractured media is always a difficult process. Unlike the case with porous media, Darcy's Law is not strictly applicable to flow through fractures, although it often can be applied empirically to large bodies of fractured rock that incorporate many fractures. Describing local flow conditions in fractured rock often poses considerable difficulty. Sowers (1981) discusses determinations of hydraulic conductivity of rock. This reference should be consulted for guidance in analyzing flow through fractured media.

3.8.1 Fine-grained sediments, such as glacial tills, are commonly fractured in both saturated and unsaturated settings. These fractures may be sufficiently interconnected to have a significant influence on ground water flow, or they may be of very limited connection and be of little practical significance.

3.8.2 Frequently, a laboratory test of a small sample of clay will determine hydraulic conductivity to be on the order of 10^{-8} cm/sec. A piezometer test of the same geologic unit over an interval containing fractures may determine a hydraulic conductivity on the order of perhaps 10^{-5} or 10^{-6} cm/sec. To assess the extent of fracture interconnection, and hence the overall hydraulic conductivity of the unit, several procedures can be used. Closely spaced piezometers can be installed; one can be used as an observation well while water is added to or withdrawn from the other. Alternately, a tracer might be added to one piezometer, and the second could be monitored. These and other techniques are discussed by Sowers (1981).

3.8.3 For situations that may involve flow through fractured media, it is important to note in permit applications that an apparent hydraulic conductivity determined by tests on wells that intersect a small number of fractures may be several orders of magnitude lower or higher than the value required to describe flow through parts of the ground water system that involve different fractures and different stress conditions from those used during the test.

4.0 CONCLUSION

4.1 By following laboratory and field methods discussed or referenced in this report, the user should be able to determine the fluid conductivity of materials used for liners, caps, and drains at waste-disposal facilities, as

well as materials composing the local ground water flow system. If fluid-conductivity tests are conducted and interpreted properly, the results obtained should provide the level of information necessary to satisfy applicable requirements under Part 264.

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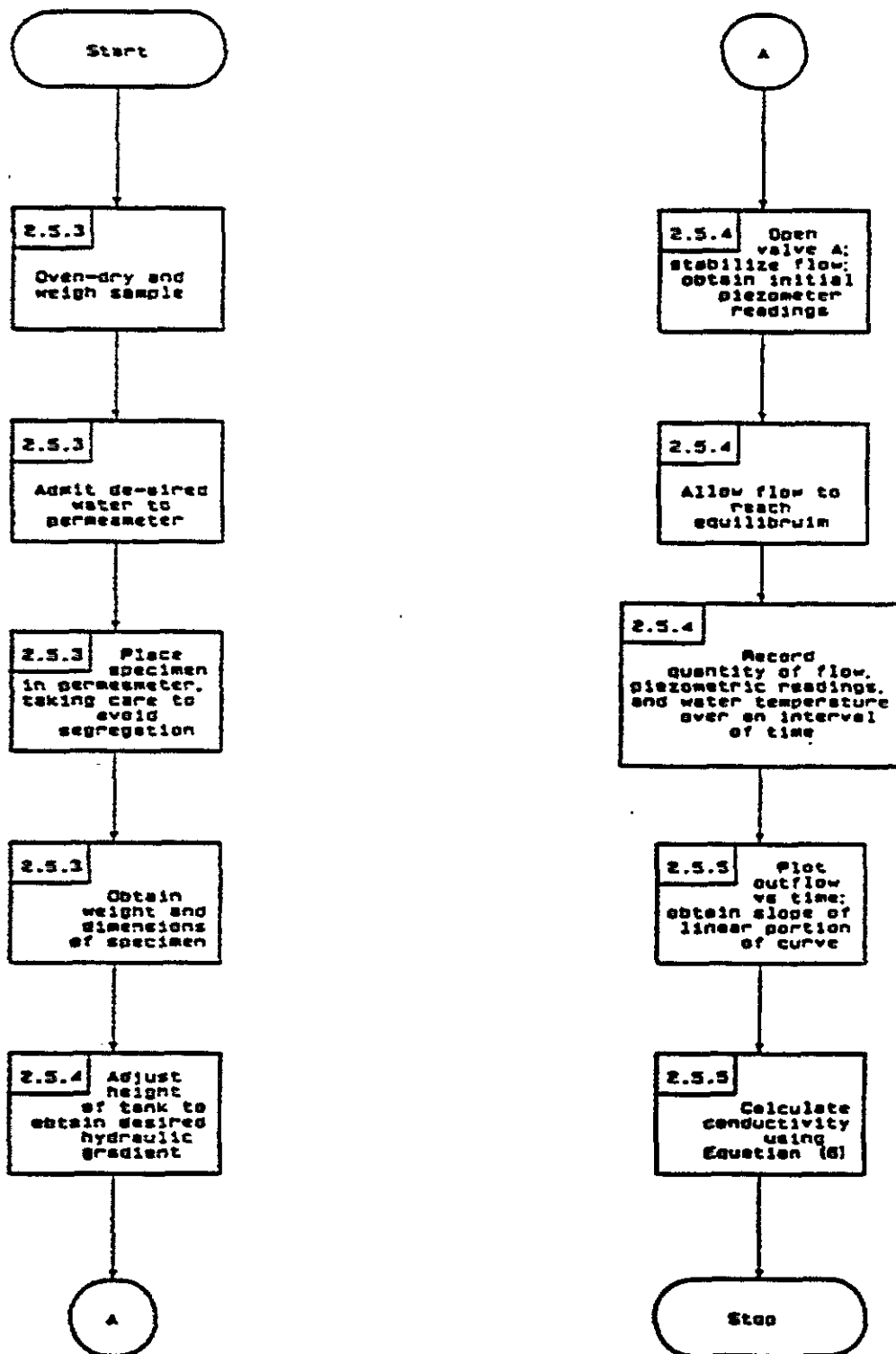
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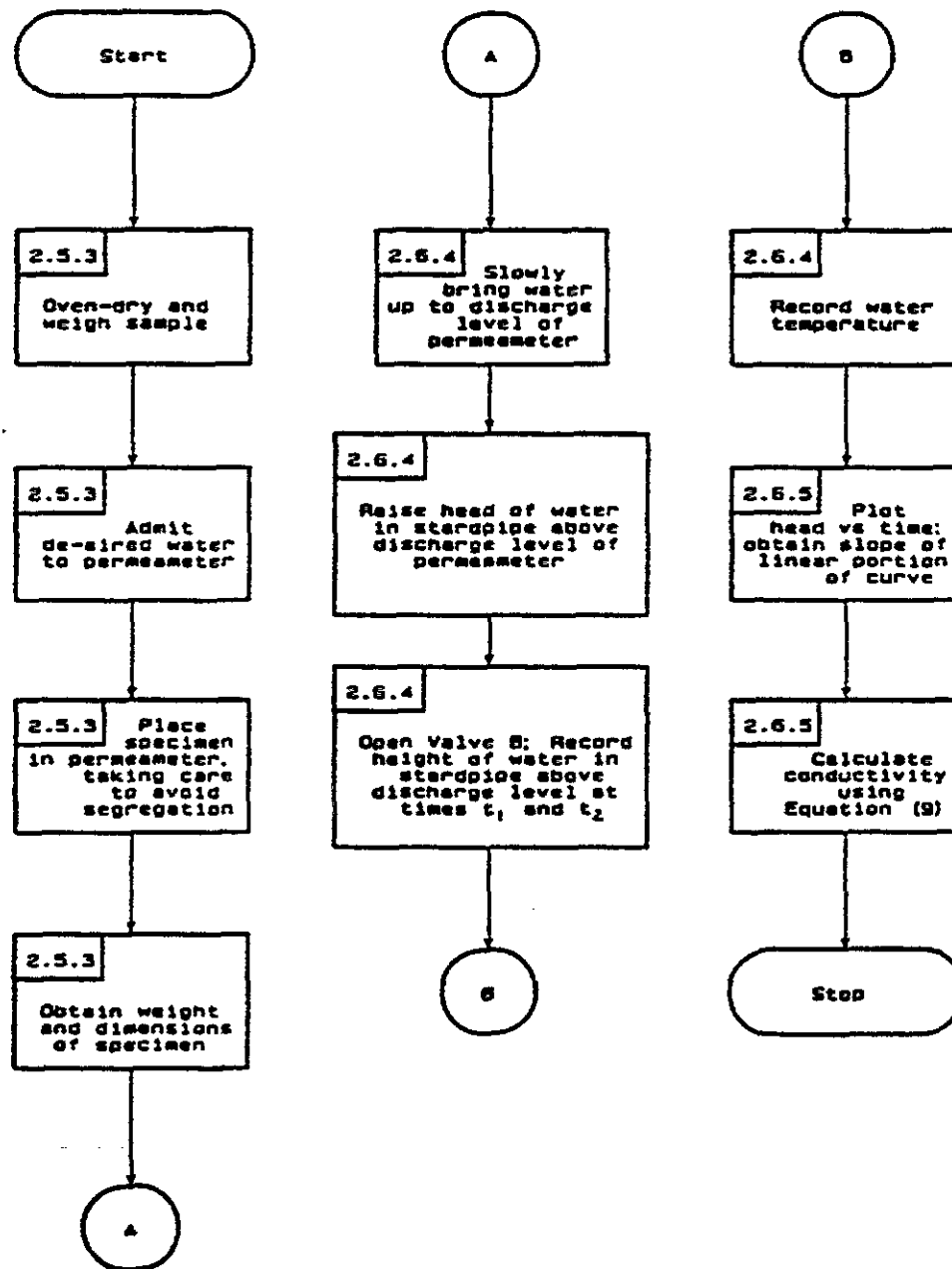
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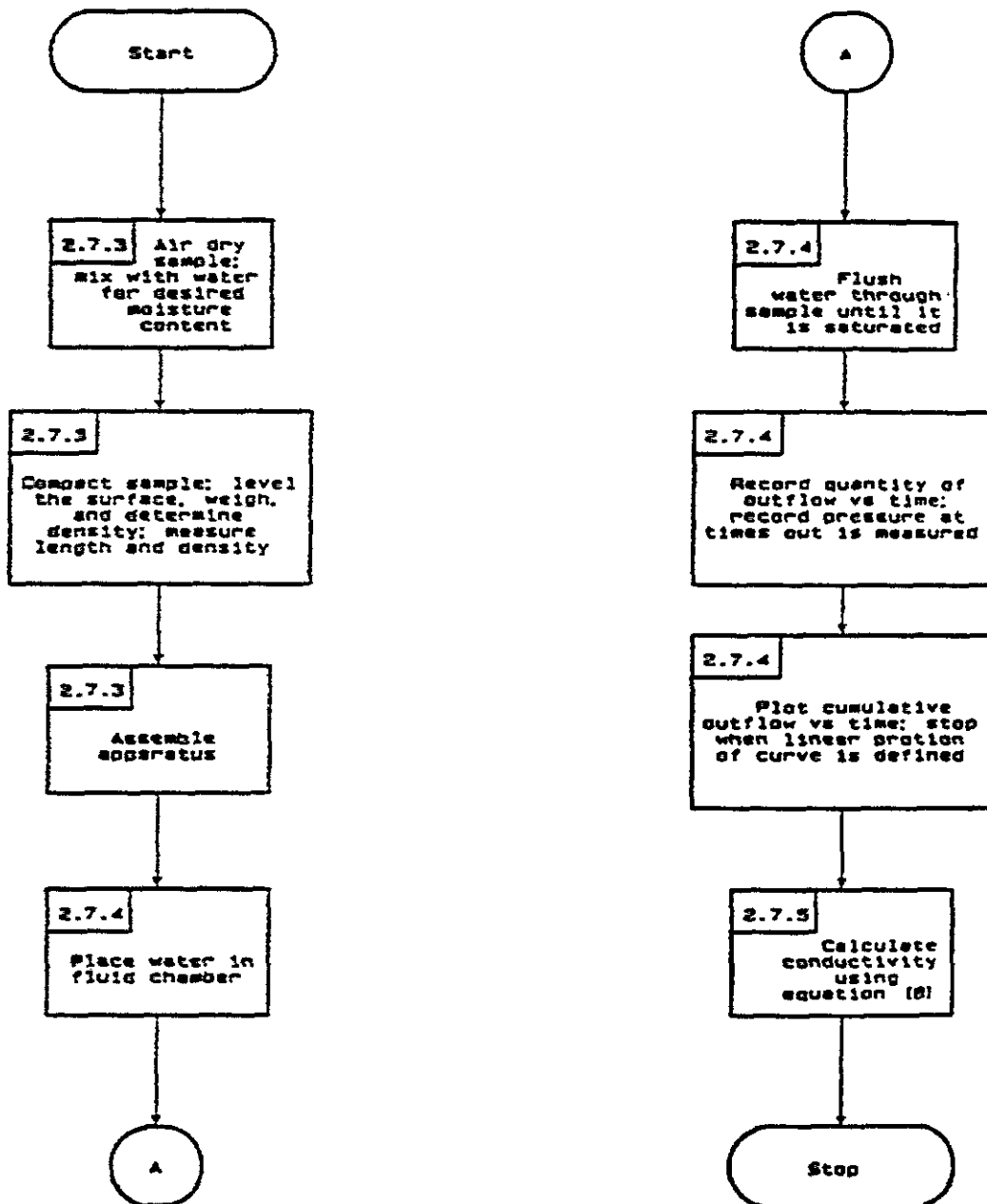
METHOD 9100 (Part)
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
CONSTANT-HEAD TEST WITH CONVENTIONAL PERMEAMETER



METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
FALLING-HEAD TEST WITH CONVENTIONAL PERMEAMETER



METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
MODIFIED COMPACTION PARAMETER METHOD

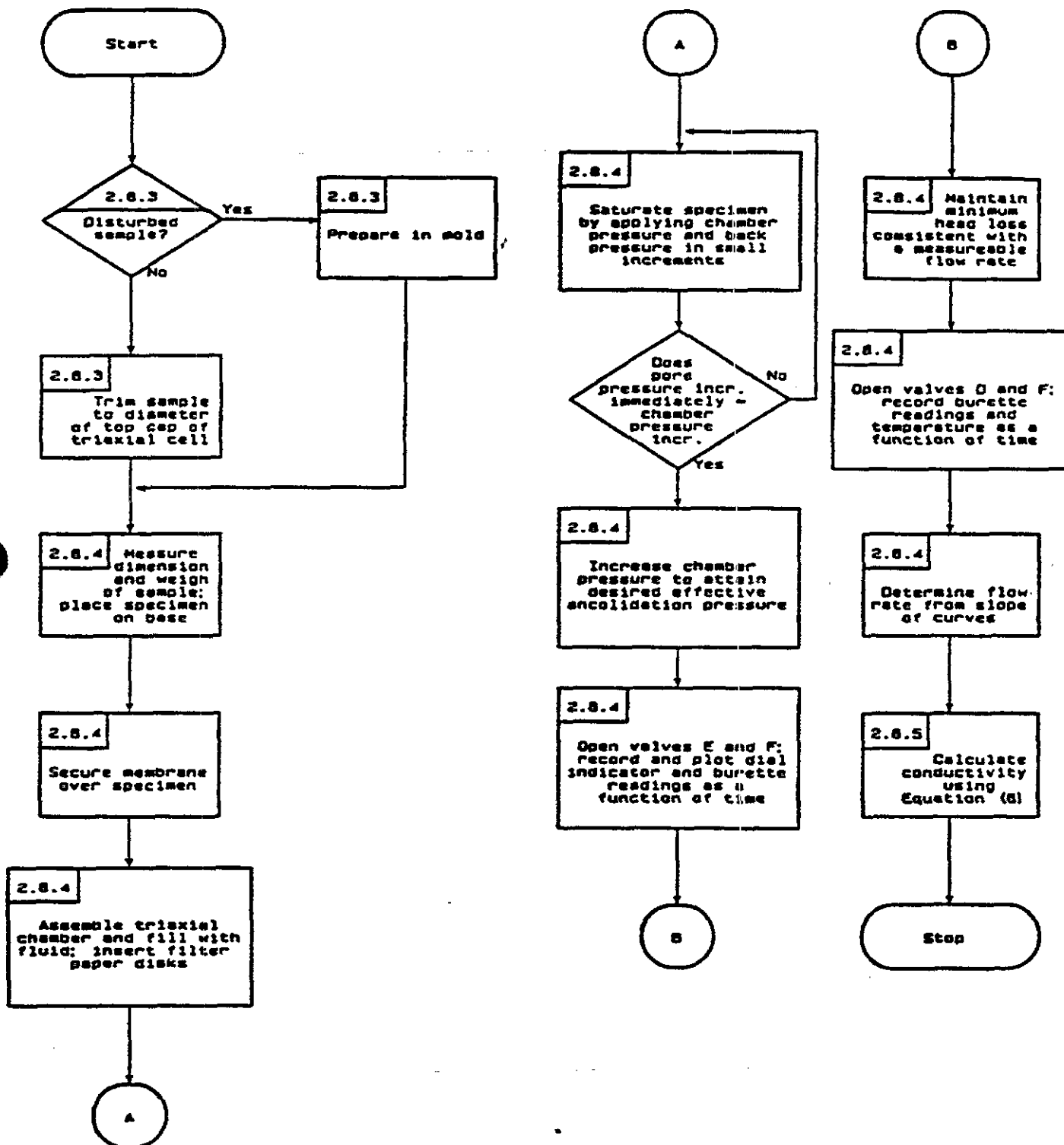


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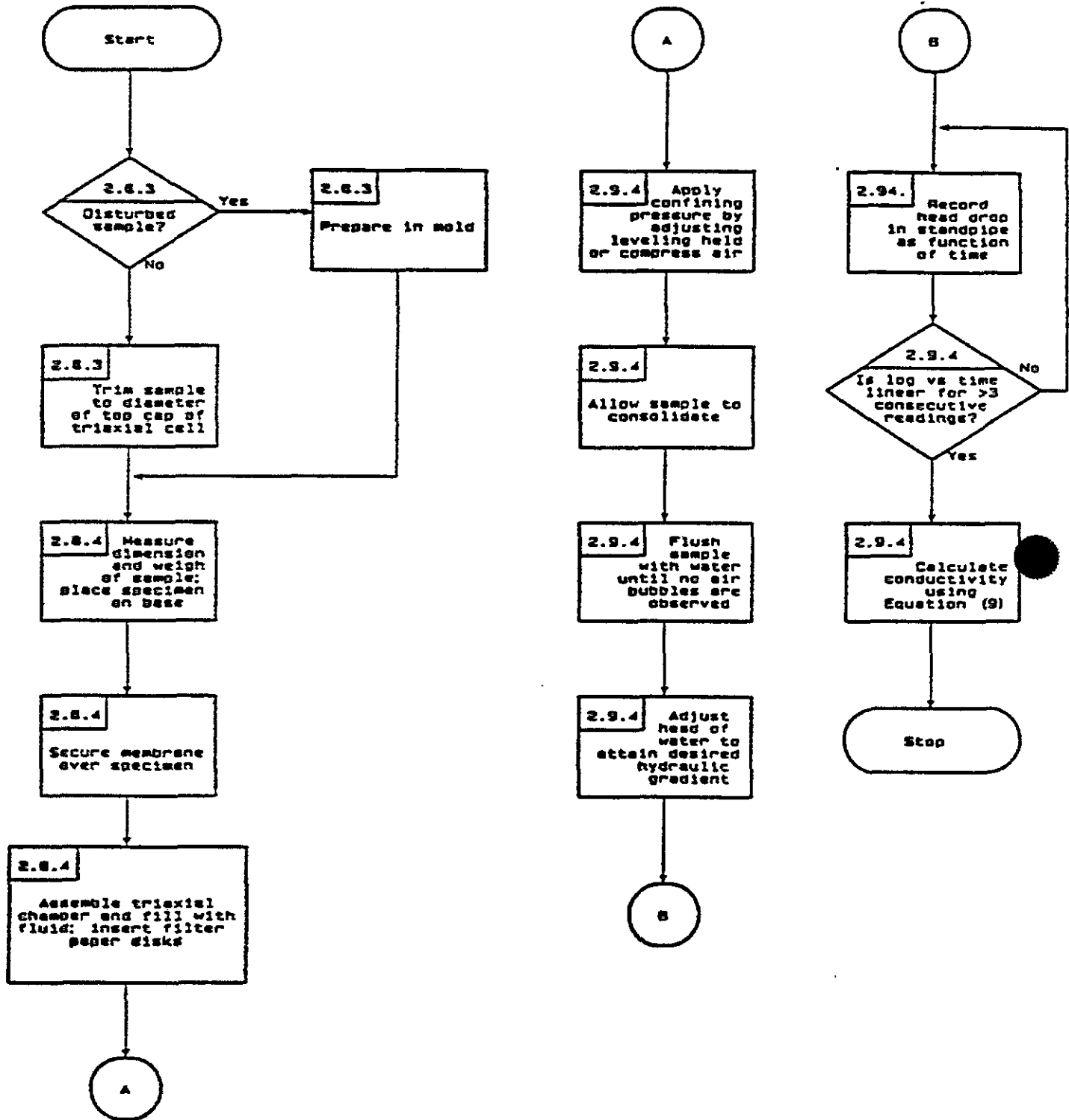
Revision 0
Date September 1986

AR302014

METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
TRIAXIAL CELL METHOD WITH BACK PRESSURE



METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
PRESSURE-CHAMBER PARAMETER METHOD



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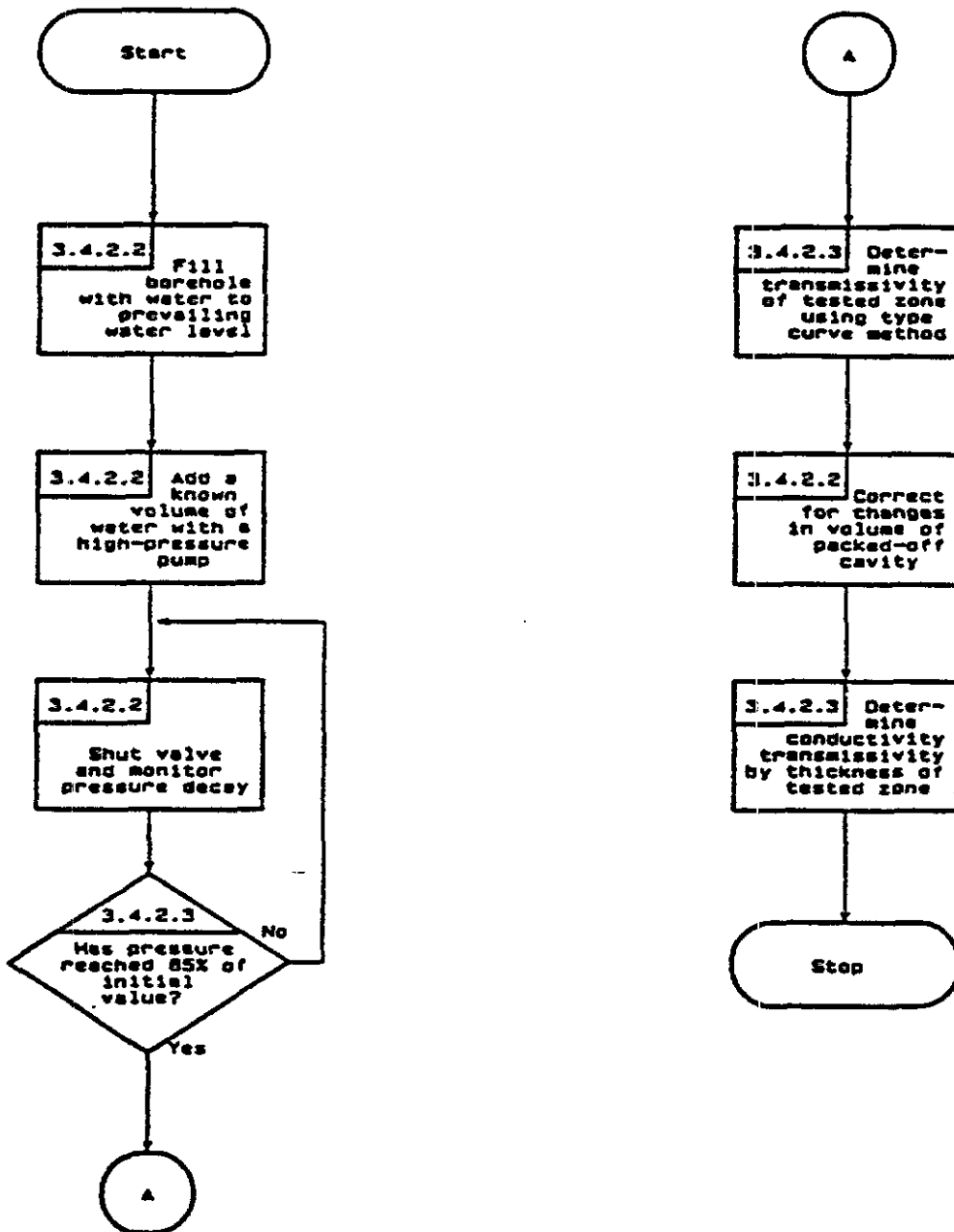
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METHOD 9100

HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:

FIELD METHODS FOR EXTREMELY TIGHT FORMATIONS UNDER CONFINED CONDITIONS

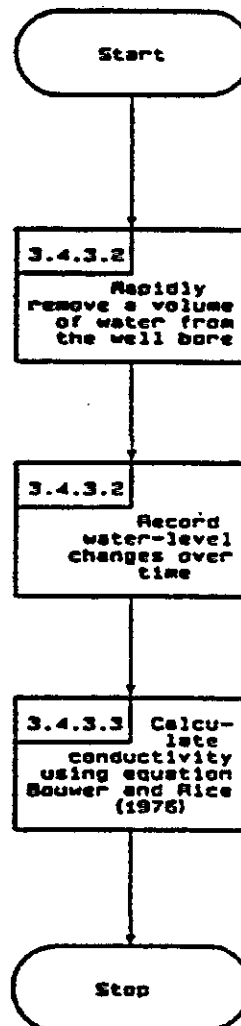


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Revision 0
Date September 1986

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METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES
FIELD METHODS FOR MODERATELY PERMEABLE MATERIALS UNDER UNCONFINED CONDITIONS

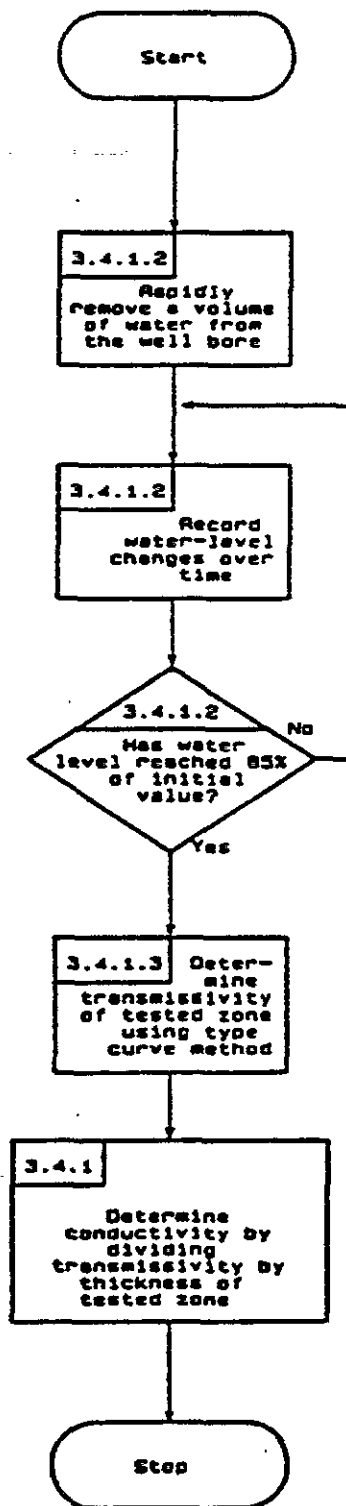


9100 - 56

Revision 0
Date September 1986

AR302018

METHOD 9100
HYDRAULIC CONDUCTIVITY OF SOIL SAMPLES:
FIELD METHOD FOR MODERATELY PERMEABLE FORMATIONS UNDER CONFINED CONDITIONS



SOP NUMBER: C.II.1

DATE: June 1990

TITLE: Field Sampling Protocol for Ambient Aquatic Chronic Toxicity Testing

SCOPE: This operating procedure describes the methods for collecting test water via grab-method for performing a static renewal chronic ambient aquatic toxicity test.

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the field.
- Serve as a means to screen for potential toxicity of the surface water to aquatic organisms.

EQUIPMENT:

- Clean teflon spoons.
- Clean leak-proof polyethylene containers for shipping of test water with labels.
- Clean containers or hoses and a pump for collection of water.
- Ice and coolers.
- Hip or chest waders.

PROCEDURE:

1. Collect a 24-hour composite of water from each pre-designated station, preferably at a period of low flow. Two liters collected every 3 hours will provide 16 liters of sample for each station.
 - o Take care not to resuspend sediments.
 - o If water depth is too shallow to collect water using a container, use an uncontaminated hose and pump. Otherwise, use a clean 1-liter polyethylene container and pour water slowly down inside of clean 5-gallon polyethylene container packed in larger container of ice.

C.II.1

AR302020

- Cap composited sample in-between collections.
 - Homogenize composite sample with a teflon spoon prior to splitting sample.
 - At least one station will be an upstream reference where no toxic impact is expected.
2. If possible, concurrently collect and analyze samples for physiochemical parameters of the water, toxicity of the test water, and contaminants of concern.
- Include field measurements of dissolved oxygen, temperature, conductivity, pH, and Eh.
 - Place split samples in clean 2-liter polyethylene containers for laboratory measurements of TAL metals, TOC, sulfide, hardness, alkalinity, and toxicity testing water.
 - Preserve samples as needed:
 - TAL metals require nitric acid to pH < 2
 - TOC requires sulfuric acid to pH < 2
 - Sulfide requires 2 ml of zinc acetate and sodium hydroxide to pH > 9
 - All samples should be kept on ice at 4°C.
3. Ship samples within 24 hours via overnight carrier (UPS or Federal Express). Samples should be packed on ice and placed in a cooler prior to shipping to the analytical laboratory.

REFERENCES: American Public Health Association, et al., 1985. Standard Methods for the Examination of Water and Wastewater, 16th edition; Part 801. Washington, DC.

Biological Monitoring, Inc., 1990. Sampling Plan, Study Design and Testing Procedures. Blacksburg, Virginia.

Ecological Analysts, August 1988. Personal communication with Wayne McCullough on SOP for Toxicity Testing, Sparks, Maryland.

U.S. Environmental Protection Agency, 1985. Short-term Methods for Estimates of the Chronic Toxicity of Effluent and Receiving Water on Freshwater Organisms, EPA-600/4-85-014. Washington, DC.

U.S. Environmental Protection Agency. Technical Support Document for Water Quality Based Toxics Control, EPA-440/4-85-014. Washington, DC.

Virginia Water Control Board, August 1988. Personal communication with Richard Ayers and Mike Shelor.

SOP NUMBER: C.II.2

DATE: June 1990

TITLE: Field Sampling Protocol for Solid-Phase Sediment Chronic Toxicity Testing

SCOPE: This operating procedure describes the method for collecting test water via grab-method for performing a static renewal chronic ambient solid-phase sediment toxicity test.

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the field.
- Serve as a means to screen for potential toxicity of sediment to aquatic organisms.

EQUIPMENT:

- Clean high-density, leak-proof polyethylene containers for shipping of test sediment with labels.
- Clean grab or core and teflon spoons to transport sediment into vessels.
- Ice and coolers.
- Hip or chest waders.

PROCEDURE:

1. Collect four liters of sediments from each pre-designated station.
 - Collect in a downstream-to-upstream manner so as to avoid resuspension of sediments that are to be sampled.
 - Collect sediments from depositional areas.
 - If possible, collect fine-particle material that is consistent with sediments found at other stations.

- Use a benthic grab or core to lift sediments up from stream bottom. A dredge is not recommended. Minimize disruption to the top layer of sediment.
 - From the grab or core, scoop the top 2 cm of sediments into vessel, thus avoiding wash-off of the top layer of sediments.
 - Collect composite sediment samples until 4 liters of sediment have been obtained from each station.
2. Avoid exposure of sediments to sunlight to avoid photolyzing.
 3. Store samples on ice (4°C) in coolers and add preservatives if necessary.
 - Sulfide will require further preservation with zinc acetate.
 - TOC must be stored in a dark brown bottle at 4°C.
 - TAL metals will require addition of nitric acid to obtain pH < 2.0.
 - If possible, concurrently sample for metals, sulfide, TOC, and toxicity testing. The pH and Eh should also be recorded in the field.
 - Split samples required for chemical analyses. One liter will be required for each of the following analyses--metals, sulfide, TOC, and toxicity testing.
 4. Ship samples on ice within 24 hours via overnight carrier (UPS or Federal Express).

REFERENCE:

Biological Monitoring Inc., 1990. Sampling Plan, Study
Design and Testing Procedures. Blacksburg, Virginia.

CH2

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(2)

SOP NUMBER: C.II.3

DATE: June 1990

TITLE: Laboratory Analysis for Solid-Phase Sediment Chronic Toxicity Testing and Ambient Aquatic Chronic Toxicity Testing

SCOPE: See Attachment, No. I

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the laboratory.
- Identify the mechanisms and materials that will ensure the proper testing for solid-phase sediment chronic toxicity and ambient aquatic chronic toxicity.

EQUIPMENT: See Attachment, Nos. IV and V

PRELIMINARY TO OPERATION: See Attachment

PROCEDURE: See Attachment

C.II.3

AR302026

**SAMPLING PLAN, STUDY DESIGN
and TESTING PROCEDURES**

Submitted to:

**Karen E.D. Seibert
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7101 Wisconsin Avenue,
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**Submitted by:
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P.O. Box 184
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June 15, 1990

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- A. Sediment Collection Methods
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- I. Daphnia Acute Test-Water
- J. Daphnia Neonate Production
- K. Fathead minnow Chronic Test-Water
- L. Artemia culture
- M. Daphnia Chronic Test-Water
- N. Hyaella Chronic test-Sediment
- O. Physiochemical Analytical Procedure for Toxicity Testing

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I. Scope and Application

The present study attempts to examine the aquatic toxicity potential of both whole sediment samples and water column samples. All sampling will be performed by staff from Dames and Moore (Bethesda, MD.) and all toxicity testing will be performed by the environmental consulting firm Biological Monitoring, Inc. (BMI, Blacksburg, VA.). Supporting chemical analyses will be performed by BMI and Environmental Laboratories, Inc. (Richmond, VA.). Both acute and chronic toxicity tests will be performed on ambient water samples from four sites using the water flea Daphnia pulex (Cladocera: Crustacea) and the fathead minnow (Pimephales promelas). Both species are sensitive freshwater indicators of potential toxicity [1]. Chronic toxicity tests will be performed in whole sediments from the same four sites using the amphipod Hyalella azteca (Talitridae: Amphipoda) and Daphnia pulex. Hyalella has been shown to be a sensitive species in both sediment and water column testing [2,3]. The following sections detail the procedures to be used in this study and specific Quality Assurance Plan which ensures that accurate data are collected and reported. A full Quality Assurance Document is appended separately.

I.1 Summary of Method

Grab water column samples are taken and composited from each site such that each water samples represent a 24 hr composite. Grab sediment samples are taken from each site as well. Sediment and water column samples are then split in order to preserve some sample from each site for subsequent chemical analyses. Samples for toxicity studies and sulfide analyses will be shipped cold (0-4°C) overnight to BMI's laboratory for immediate testing. Details of sampling and sample preparation are described in Section III.

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Whole samples (no dilutions) will be used in testing. In addition to the four sites being tested, controls will be simultaneously tested consisting of known, clean dilution water or sediment. Multiple test organisms are exposed in replicate treatments in all tests such that valid effect levels can be determined. Details on test organism history and handling are presented in section IV. Sediment testing utilizes sediment samples from each site (and controls) and BMI standard dilution water. In this way, we can partition the toxicity effects of the sediment alone. The water column tests utilize ambient water samples (and controls) which enables us to determine the degree of toxicity associated with the water alone. Chemical analyses performed along with the biological testing will enable us to determine the relative contributions of certain heavy metals (chromium, arsenic, and copper) to overall observed toxicity. Details of the testing and analytical procedures are described in section V. Data handling and analyses are discussed in section VI.

II. Safety Precautions

As in all sample handling and testing performed by BMI, latex gloves are worn when handling samples and goggles and lab coats are worn when testing samples. If sediment or water samples reveal particularly noxious fumes or odors, all testing and sample handling is performed in an explosion-proof fume hood equipped with a 1 hp. fan and vent system. All other sample handling and testing is performed in a temperature-controlled laboratory equipped with a constant ventilation system to remove odors and purify the air.

III. Sample Collection and Processing

III.1 Sediment Samples

A 4 liter sample of sediment is collected at each site which is then put into a high density polyethylene container and mixed on-site using a teflon spoon. The sample is then split into four one liter portions for subsequent chemical analyses and toxicity studies. Samples for sediment sulfide will be preserved separately with zinc acetate [4]. Samples for total organic carbon will be stored in a dark bottle and kept cold (0-4°C). Details of sediment collection methods are described in Appendix A. All sediment samples will be shipped via overnight courier in sealed containers on ice to BMI for processing.

Once received by BMI, sample containers are externally examined, and sample temperature measured in order to assess sample integrity during shipment. Any abnormalities are recorded by the laboratory scientist on the chain-of-custody form. Samples are then processed according to the analyses to be performed. In this case, samples to be analyzed for metals will be checked to make sure the pH $\leq$ 2.0 as per the preservation protocol. If not, the sample will be declared invalid and a second set of samples will be necessary. If the pH is $\leq$ 2.0, these samples will then be analysed by staff at Environmental Laboratories, Inc. along with samples for total organic carbon. All samples include tracking ID numbers, and custody sheets. Metal analyses will include total chromium, hexavalent chromium, copper, and arsenic. All metals will be analysed as total recoverable. Sample analyses for sulfides and toxicity studies will be initiated by BMI on the day they are received.

III.2 Water Samples

Water samples from each site will be manually composited over a 24 hr period by taking 2 liter samples every 3 hours. This will yield a total volume collected of 16 liters over the 24 hr period. Samples will be collected using a clean 1 liter polyethylene container and poured slowly (to minimize possible volatilization of chemicals) down the side of a clean 5 gallon polyethylene container which is packed in ice in a larger container. The 5 gallon container is closed to air between sampling times and insulated to keep temperature $\leq 4^{\circ}\text{C}$.

Following the 24 hr period, each sample will be homogenized by stirring the sample with a clean teflon spoon. Two 2-liter aliquots will then be poured into separate clean polyethylene cubitainers for processing and analyses. One aliquot will be preserved with nitric acid to a pH ≤ 2.0 for metal analyses and labeled. The second aliquot will be labeled for toxicity studies. All samples will be handled and shipped as per the sediment samples in III.1 above. Laboratory processing will also be identical to that described for sediment samples above. Standard laboratory procedures for all sampling and sample processing are described in Appendix B.

IV. Culture Methods and Materials

IV.1 Test Organism Cultures

Daphnia pulex, fathead minnows, and Hyaella azteca are all cultured at the BMI laboratory in Blacksburg, Virginia. Daphnia pulex originated from a strain received in 1983 from the U.S. EPA Athens, Georgia laboratory. Although fathead minnows are continuously cultured at BMI, minnow adults (breeders) are periodically obtained from other sources including U.S. EPA Cincinnati, Ohio laboratory, Florida Biosystems Laboratory (Gainesville, Florida), and SP Engineering, Inc. (Amherst, Massachusetts). This ensures that the minnow population is outbred diversifying the gene pool of fish used in testing. Hyaella were originally obtained from Carolina Biological Supply Co. (Greensboro, N.C.).

Specific methods relating to the culture of Daphnia pulex, fathead minnows, and Hyaella are presented in Appendix C, D, and E, respectively. All cultures at BMI are routinely challenged with reference toxicants such as sodium chloride, cadmium chloride, and sodium lauryl sulfate. All reference toxicants are laboratory reagent grade chemicals ($\geq 99\%$ pure) which are monitored by batch number and expiration date. Details of reference toxicant procedures and quality control procedures relating to organism culture are presented in appended Quality Assurance Document.

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IV.2 Culture and Dilution Water

All three test organisms are cultured and tested using BMI standard dilution water. This water consists of dechlorinated (activated carbon), filtered (0.2 g glass filter), and aged (aerated for at least 48 hrs prior to using) Blacksburg municipal water. A full priority pollutant scan is performed semiannually on this water and daily physiochemical measurements (including total residual chlorine) are also performed in order to ensure acceptability of this culture and dilution water. The water does not come in contact with any metal in the laboratory except for stainless steel valves and fittings to ensure no metal contamination. The most recent chemical analysis on this water is shown in Appendix F. BMI standard water is characterized as a relatively soft, low alkalinity water, moderate pH, with little or no solids or organic carbon. This water source has been the primary culture and test dilution water at BMI for the past 8 years, attesting to the constancy and acceptability of its quality. A more detailed description of dilution water handling is presented in Appendix F.

IV.3 Culture and Test Materials

Appendix G lists the equipment and facilities at BMI related to toxicological testing. All three species utilized in this study are cultured in glass containers (aquaria, dishes, beakers, etc.) which undergo rigorous periodic cleaning by hand (see Appendix G for details). Test containers for these species include glass beakers, dishes, or aquaria depending on the study design. Specific details are presented in subsequent sections on test methods.

Aeration to cultures and test chambers when necessary, are supplied via a centralized air compressor system equipped with a nylon fiber filter. Air is transported to various points in the laboratory via plastic pipe and tygon tubing.

Laboratory temperature is maintained at $20 \pm 2^{\circ}\text{C}$ via thermostatically controlled heater/air conditioner system. Daily maximum and minimum temperatures are recorded in a dedicated log book. For cultures or tests requiring different temperature regimes, BMI has more than 300 square feet of water bath space available equipped with automatically controlled heater or chiller units.

Lighting is maintained on a 16:8 hr light: dark photoperiod via automatic timer. Lighting is fluorescent and is between 75 and 100 foot/candles on the laboratory counters where test chambers are set. This is the recommended photoperiod and light intensity for organism culturing and toxicity testing [5,6]. The photoperiod and light intensity are checked at least quarterly and adjusted if necessary. All checks are recorded in a dedicated notebook for this purpose.

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V. Test Methods and Materials

V.1 Water Column Testing-Acute

Protocols for the fathead minnow and Daphnia pulex water column acute tests are presented in Appendices H and I, respectively. A 48 hr exposure period is proposed for both species consistent with U.S. EPA acute toxicity test guidelines [5]. The acute tests are performed in a static mode and organisms are not fed during the test. Juvenile fathead minnows (5-30 d old) and Daphnia pulex neonates (< 24 hr old) are used in testing. Procedures for obtaining Daphnia pulex neonates are presented in Appendix J. The tests are performed at $20 \pm 2^\circ\text{C}$.

Ten organisms are utilized in each of two replicate test chambers per treatment. One liter glass dishes (800 ml test volume) are used for fathead minnows and 300 ml glass dishes (100 ml test volume) are used for Daphnia pulex. Since ambient samples from each site are the treatments in this study, no dilutions of the samples are tested. Organisms are checked during the first 6 hours of exposure and thereafter at 24 and 48 hrs. Dead organisms, or those not responding to gentle prodding, are removed from the test chamber and recorded on dedicated log sheets.

The number dead and the number of organisms effected (littleness, erratic movement, and other abnormal behavior) are tabulated and analyzed at 24 and 48 hr of exposure. Data are entered and analyzed on an IBM AT microcomputer equipped with a versatile data analysis program (Toxstat, Dr. Gulley, Univ. of Wyoming - version 3.0). This program computes various statistics depending on the nature of the data. All computer printouts, bench data sheets, and QA/QC data are presented in the report. Section VI details the analytical methods used. The attached Quality Assurance Document details procedures for testing, data collection, and data reporting used by BMJ.

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V.2 Water Column Testing - Chronic Tests

Fathead Minnow

Details of this test procedure are presented in Appendix K. This is a 7 day growth and survival test conforming to U.S. EPA test guidelines [6]. The test is performed in a static/renewal mode, in 600 ml glass dishes (500 ml test volume) at $25 \pm 1^\circ\text{C}$ using a water bath and temperature controller. The test is started with newly hatched larvae (< 24 hr old) which are fed live Artemia nauplii (Appendix L) three times daily. Test solutions are renewed daily using fresh samples from each site. The test endpoints are dry weight and survival for each replicate chamber. The test is performed in triplicate for each sample, 10 fish per replicate. Growth and survival data are analyzed using the same computer program as indicated in section V.1.

Daphnia pulex

The Daphnia chronic test procedures are detailed in Appendix M. This is a 21 day test conforming to U.S. EPA test procedures under Toxic Substance and Control Act guidelines [7] and ASTM E-47 subcommittee on Aquatic Toxicology standards [8]. The test examines survival and reproduction of Daphnia pulex over the 21 day exposure period. Testing is performed in a static/renewal mode at $20 \pm 2^\circ\text{C}$ in 100 ml beakers (80 ml test volume) with renewals performed 3 times weekly. Offspring are counted daily and removed. Number of offspring and number of surviving adults per chamber are analyzed using the same EPA computer program described above.

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V.3 Sediment Chronic Toxicity Testing

Hyalella azteca

Methods for this test are described in Appendix N. These procedures conform to the recently approved ASTM E-47 standard practice for this species [9]. The test is performed at $20 \pm 2^\circ\text{C}$ and extend for 30 days. The test is performed in a static mode with no renewals and organisms are fed three times weekly. The test is started with 24-48 hr old Hyalella and measures growth, survival, and incipient reproduction. The test is performed in 800 ml beakers containing 100 g of sediment and 400 ml of BMI diluent. Test endpoints are analyzed using the same EPA computer program.

Daphnia pulex

Methods for this test are essentially the same as those described in section V.2 and Appendix M with the following exceptions: (1) The test chambers are 800 ml beakers containing 100 g of sediment and 400 ml of BMI diluent and (2) No solution renewals are performed during the test. Test duration, conditions, endpoints, and analyses are otherwise identical to those described for Daphnia pulex water column chronic toxicity testing.

V.4 Chemical Analyses

Analyses for Toxicity Studies

Concurrent with each toxicity test, measurements of temperature, pH, conductivity, alkalinity, hardness, and dissolved oxygen are made daily in each test chamber. Details of the procedures used are presented in Appendix O. Each analytical method conforms to Standard Methods [4] and U.S. EPA Standard Good Laboratory Practices [10]. Quality assurance procedures for these analyses are included in Appendix O and the adjoining Quality Assurance Document.

Other Chemical Analyses

Heavy metals and total organic carbon will be analysed by Environmental Laboratories, Inc. (Richmond, Virginia) which is an EPA certified laboratory and well equipped to perform these analyses. All metals will be analyzed using graphite furnace atomic absorption spectrophotometry (Perkin-Elmer HGA-600) with appropriate use of blanks and standards. In addition, frequent spiked samples will also be performed for quality assurance purposes. Each sample will be analyzed in replicate and occasionally triplicate to ensure accurate analytical data. All quality assurance data are reported along with the sample results. Total Organic Carbon will be measured by Environmental Laboratories, Inc. using a combustion-infrared carbon analyzer [4]. Standards using dried potassium biphtbalate will also be performed to ensure accurate data.

Total sulfide will be measured in sediment elutriate samples using the Iodometric method [4]. Standards will also be analyzed and reported along with the sample data.

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VI. Data Analyses and Reporting

All data from toxicity studies are input and analyzed using an IBM AT microcomputer and the data analysis program "Toxstat" (Dr. Gulley, Univ. Wyoming vers. 3.0). The program handles data entry, file manipulations, and various statistical analyses. All analyses include the input data along with the statistical analyses for error checking and validation of analytical results.

Since whole samples without dilutions will be used in this study, continuous end-points such as LC50s or NOECs (no observed effect concentrations) can not be calculated. Instead, intersite comparisons will be derived using two way ANOVA (site and replicate number) followed by a posteriori means tests such as Dunnetts and Shapiro-Wilks or multiple comparisons such as Tukeys Test depending on the data being analyzed and the conformance of the data to parametric analyses. Tests for homogeneity of variances (Bartlett's Test) and normality (Chi-Square) are performed on a given data set prior to analysis. These analyses are printed and reported along with the statistical test results. Significance is determined at $p = .05$ level in all analyses. Treatment comparisons which have a probability greater than 0.05 of meeting the null hypothesis (no difference between control or reference and a given site) are considered non-significant differences and therefore the same. Quality Assurance relating to data handling, reporting, and storage, are included in the accompanying Quality Assurance Document.

VII LITERATURE CITED

1. U.S. EPA. 1990. Technical Support Document for Water Quality-Based Toxics Control. Office of Water and Standards, Washington, D.C. Draft.
2. Nebeker, A., M. Cairns, J. Gabsletter, K. Malneg, G. Shuytema, and D. Krawczyk. 1984. Biological Methods for determining toxicity of contaminated freshwater sediments to invertebrates. *Envir. Toxic. Chem.* 3:617-630.
3. Ingersoll, C. and M. Nelson. 1990. Testing sediment toxicity with Hyalella azteca (Amphipoda) and Chironomus riparius (Diptera). ASTM, STP, 13th symposium on Aquatic Toxicology and Risk Assessment. In Press.
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7. US EPA. 1985. Office of Toxic Substances, Federal Register CFR 40:797.1300.
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9. American Society of Testing and Materials. 1990. Standard Guide for conducting sediment toxicity tests with freshwater invertebrates.
10. US EPA. 1978. Quality Assurance guidelines for Biological Testing. EPA-600/4-78/043. ESML Las Vegas, Nevada.

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Field Collected Test Sediment

A benthic grab will be used rather than a dredge to minimize disruption of the sample (see ASTM Guide for Sediment Collection, Storage, Characterization, and Manipulation and ASTM Standard Guide D 4387). Sediment will be collected from the upper 2 cm since this is often determined to contain the most potential toxicity. This operation is facilitated by having the grab opened from the top so that the undisturbed sediment surface is exposed. The sample is transferred to a labeled clean sample container. To guard against the possibility that contaminants associated with sediments include compounds that readily photolyze, we will minimize direct sunlight during collection. All sediment samples will be split in the field for chemical and biological analyses. One portion of each sample will be placed in a container containing nitric acid to a pH <2.0 for subsequent metals analyses (EPA, 1979 EPA-600/4-79-019). A second portion will be placed in a separate container for sulfide analyses. The third portion will be cooled to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in the field using ice and/or ice packs and insulated coolers for toxicity studies and total organic carbon. Each sample is given a clearly labeled sample ID number and a chain-of-custody sheet is completed for each sample by the sampler. A sample chain-of-custody form is appended.

Sediment samples are shipped overnight at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and will be utilized in testing immediately upon arrival to the lab (within 36 hrs of collection). Sediment will be stored in sealed, high density polyethylene containers which are of suitable quality for this purpose. It is desirable to avoid contact with metals, including stainless steel and brass sieving screens. The samples will be thoroughly mixed and wet-press sieved to remove large particles and endemic organism, especially predators. Depending on its density and composition, sediment may be diluted and mixed with 1:1 overlying water to facilitate sieving.

This is reported if necessary. Qualitative descriptions of the sediment will include color, texture, presence of macrophytes, animals, tracks, and burrows.

The natural geochemical properties of test sediment collected from the field must be within the tolerance limits of the test species. Ideally, limits for the test species should be determined experimentally in advance. If not, factors such as particle size and distribution, organic carbon content, pH, etc. will be run concurrently with testing to determine if the limits are exceeded in the test sediments.

BMI BIOLOGICAL MONITORING, INC.

P. O. Box 184 • Blacksburg, Virginia 24060 • Telephone 703-953-2821

BIOASSAY SAMPLE COLLECTION AND CHAIN OF CUSTODY FORM

THIS SECTION TO BE COMPLETED BY PERSON COLLECTING SAMPLE (SAMPLER)

Client's Name: _____

Sampler's Name: _____ Affiliation: _____

Sample Source: _____ NPDES Permit#: _____

Description of Sampling Site: _____

Description of Sampling Equipment and Methods: _____

Sample Type:

Grab: Collected-Date _____ Time _____ Initials _____

Composite: Collected-From Date _____ Time _____

To Date _____ Time _____ Initials _____

Volume Collected: _____ Container Type: _____

Is This Sample Chlorinated? (check one) No ☐ Yes ☐

Date and Method of Shipment to BMI: _____

How Was Sample Packed for Shipment: _____

Type of Tests to be Performed: _____

Comments: _____

I certify that the above information is correct. _____

Sampler's Signature

Date

FOR BMI USE ONLY

BMI Sample ID# _____ Received by: _____

Upon Arrival at BMI: Date _____ Time _____

Sample Temperature _____ °C Sample pH _____

Comments: _____

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STANDARD OPERATING PROCEDURES

A.8 Test Scheduling and Client Contact

1. When a test is to be scheduled, a date is chosen which is convenient for the client for sampling and shipping.
2. The client is informed of the sampling and test dates (test date is the date sample is received).
3. The scheduler sends cooler(s), container(s), Sample Handling, Collection and Shipping Instructions (Fig. 4), and Sample Collection and Chain of Custody Form (Fig. 5) to the client or sampler.
4. When a test is subcontracted and the client does not collect the sample, the subcontractor must be contacted about the sampling date, and inquiries about any needed sampling materials are made.
5. The scheduler fills out a Scheduling and Invoice Form (Fig. 6), including any extra instructions relevant to the project scientist. (The project scientist will be assigned at this point or at the time the sample is received by the lab manager.)
6. The test date is marked on both schedule calendars - one in the lab, and one in the lab manager's office.
7. The lab manager is made aware of any special arrangements for a specific test by the scheduler as soon as the circumstances are known.

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Sample Collection, Handling, and Shipping Instructions

Biological Monitoring, Inc. (BMI) conducts bioassays in conformance with appropriate U.S. EPA specified guidelines. Within these guidelines, proper effluent sample collection and handling procedures are set forth. BMI provides its bioassay clients with the following information to assure sample integrity. The following procedures must be adhered to so that sample collection and handling procedures do not invalidate test results.

Sampling Point and Sample Type

Ordinarily, the effluent sampling point and sample type (grab or composite) are specified in the NPDES discharge permit. In most cases, it is desirable to sample at the outfall or, if the effluent is chlorinated, just prior to chlorine contact. If there are questions concerning the sampling point, or sample type, the regulatory agency should be contacted.

Sampling Equipment and Sample Containers

All equipment coming into contact with the effluent sample must be clean glass, teflon, or polyethylene which has been rinsed at least three times with effluent. If an automatic composite sampler is used, the sample must be cooled during collection. Sample containers can be either glass or polyethylene. If properly cleaned, glass may be reused. Polyethylene containers must be discarded after use.

Sample Handling

Samples collected for toxicity testing must be immediately placed on ice, and must be shipped in an insulated container or cooler with ice or cold packs. Upon arrival at BMI the sample is stored in a refrigerator (4°C) until used. Testing must be initiated within 36 hours of sample collection. Therefore, the sample must be shipped via a reliable overnight parcel service or hand delivered to BMI immediately after collection.

Sample Volume

For each bioassay, a 1 gallon (3.8 liter) sample of effluent is usually sufficient. This does vary with test species and with organism size and age. Therefore, if questions exist regarding the required volume, BMI should be contacted.

Sample Collection Documentation

For each effluent sample collected and shipped to BMI, a BMI Bioassay Sample Collection and Chain of Custody Form must be completed. This form, which is signed by the sampler, documents the sampling collection and handling procedures which were followed and acts as a permanent record for that sample. Should any questions arise concerning effluent sampling and handling, BMI would be happy to assist.

SHIPPING ADDRESS: Biological Monitoring, Inc.
Route 460 South
Blacksburg, Virginia 24060

MARK ALL SHIPMENTS: Attention - Laboratory Manager

BIOASSAYS SCHEDULING & INVOICE GENERATION FORM

Today's Date: / / Company:..... Prefix:.....

Contact's Name: Title: PH. ().....

Mailing Address:.....

.....

.....

Shipping Address:.....

.....

.....

Containers To: Data To:.....Invoice To:.....

BMI Project #: Clients P.O. #:

Services Scheduled:

Starting Date:

Description:

Price:

Rate:

Regular

Emergency (+50%)

Other

Special Conditions:.....

.....

Collection Package needed: Y / N Ship By: / / Date Shipped: / / Int.

Collection or pickup Needed: Y / N Collection Date / /

Delivery or Service Date: / /

Comments:

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

A.9 Sample Check-in, Storage, and Disposal

1. Upon arrival or initiation of a project, and immediately after removal from coolers, each sample is checked in on the accompanying Sample Collection Form by receiving a unique identification number, checking temperature, pH, conductivity and salinity. Date, time of arrival and technician's signature are also recorded.
2. The sample identification number consists of a prefix unique to each client plus a six-digit date and a number indicative of multiple samples received from the same client on the same day. (Example: BMI040489-1, BMI040489-2). The sample identification number is marked on the sample container.
3. The completed schedule invoice form is obtained from the lab manager's office, and sample information is recorded on the Project Progress Log Sheet (Fig. 7), also found in the office of the lab manager.
4. If test initiation procedures are not scheduled to begin upon receipt of the sample, it is stored at 4°C.
5. When a sample is no longer needed, it is disposed of in a manner appropriate for the particular substance.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

A.10 Project Progress and Data Processing

1. Upon arrival of a sample or initiation of a project, information regarding the project is recorded on the Project Progress Logsheet (Fig. 7). This includes the client's name, project description (specific tests requested) and sample name.
2. Each sample is given a sample ID number and is assigned a project scientist and a quality control officer. A Laboratory Work Order (Fig. 8) is filled out by the laboratory manager.
3. Upon completion of the required test(s), data and/or reports are prepared by the project scientist and reviewed by the quality control officer.
4. Results should include raw biological data in tabular form, including daily records of affected organisms in each concentration, table of LC50's, NOEC's, etc., summaries of physical and chemical data and any pertinent QA data.
5. The dates that the results and invoice are mailed out are also noted.

[illegible]

6/97

BMI BIOLOGICAL MONITORING, INC.

P. O. Box 184 • Blacksburg, Virginia 24060 • Telephone 703-953-2821

Laboratory Work Order

Assigned to:.....

Client:.....

Sample ID#.....

Work Description:.....

Test Conditions: Organism(s) and age-

Test Mode-

Test Duration-

Dilution Water-

Concentrations-

Replicates-

Temperature-

Feeding-

Special Conditions:

Initiation Date:.....

Completion Date:.....

Assigned by:.....

Date:.....

Fig. 8

BMI/SOP3.0

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8-8

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BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

D.1 Daphnia pulex Stock Cultures

1. Daphnia pulex stock cultures are maintained in glass "battery" jar aquaria with dechlorinated tap water as culture media.
2. Cultures are held at 20°C ($\pm$ 2°C).
3. Feeding routine requires that each culture be fed 4 ml of Daphnia food (see SOP F.2 for directions on preparing this food) three times per week. Designated feeding days are included in the Routine Laboratory Procedure Checklist (Fig. 1).
4. Cultures are renewed once a month on a rotating basis so that some mature cultures are available at all times. Renewing a culture involves discarding the old culture, and starting a new culture with a clean battery jar, dechlorinated tap water, and 7 to 12 pregnant healthy organisms. Cultures should be fed at the time of renewal. Label the battery jar with the date of renewal.
5. Mature cultures should be thinned as needed to avoid overcrowding. While thinning, or once per week, 50% of the culture water should be renewed.
6. Cultures showing signs of stress, such as the presence of ephippia (an opaque black spot on the end opposite the head) should be renewed immediately.
7. All information and observances should be recorded on the Cladoceran Log Sheet (Fig. 13) designated for each culture.

BMI Laboratory-Cladoceran Log Sheet

☐ Daphnia magna ☐ Daphnia pulex ☐ Ceriodaphnia dubia ☐ Other _____

Rank # _____ Feeding Regime _____ Log Sheet Started _____ 19 _____

[illegible]

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

C.0 Fish Culture and Maintenance

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BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

C.1 Fathead Minnow Cultures

1. Culture tanks currently being used at BMI consist of 10 and 20 gallon long glass aquaria equipped with undergravel filters, bubble-up corner filters, heaters, thermometers, and small gravel as substrate. Dechlorinated tap water is used as media.
2. Spawning substrates placed in each tank to provide a surface for eggs are 4 to 5 inch sections of 4 inch PVC pipe cut in half lengthwise. There should be as many substrate sections as male fatheads in each culture tank.
3. To set up a new tank, rinse all clean components with dechlorinated water, assemble under gravel parts, and add 3 inches of rinsed gravel and dechlorinated water to fill. Assemble bubble-up filter adding 1 - 2 inches of rinsed carbon and ammo-chips and filter fluff. Attach airline to columns and bubble-up filter and adjust for moderately heavy air flow.
4. Heaters are adjusted to maintain water temperature of 25°C (± 2°C).
5. Photoperiod to encourage spawning is 16 hrs light and 8 hrs dark.
6. After checks on chlorine (should be < .01 mg/L) and temperature (25°C), one or two fish can be added to encourage growth of bacteria in the substrate. No more fish should be introduced for 2 - 3 weeks depending on water quality (nitrite levels should remain below .01 mg/L; ammonia levels below .1 mg/L).
7. Ideally, 1 male and 2 females for 10 gallon aquaria and 2 males and 4 females for 20 gallon aquaria should be maintained. By identifying banded males and removing them, remaining fish can be identified and the male/female ratio adjusted.
8. Tanks are kept clean by periodic siphoning and replacing part of the water according to the Laboratory Procedure Check List. Nitrite, ammonia, DO, and pH are measured and logged according to this schedule also.
9. Fish are fed frozen brim shrimp (thawed) in the morning and Tetramin in the afternoon and on weekends. Care should be taken not to overfeed.
10. Periodically (about twice a year) tanks should be broken down and cleaned

by first removing fish and water, then scrubbing aquarium glass and filter parts with no soap, and rinsing the substrate with tap water. Finally, proceed with steps to set up a new tank as explained in #3.

11. In the event that diseased fish are present in an aquarium, the fish must be destroyed and the aquarium, filter parts, and substrate soaked in a 5% chlorine bleach solution for 24 hours. After soaking, pour out bleach solution, rinse well with tap water and allow to air dry for 48 hours before setting up tank again.
12. All information pertaining to culture tank conditions, water quality and stocking must be recorded on the Fish Culture Tank Log Sheet (Fig 11). Records are kept of spawning frequency and batch size.

Fish Culture Tank Log Sheet

A.M. _____

P.M. _____

Weekends

Tank #:

Match #:

Species: _____

[illegible]

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

C.2 Fathead Minnow Egg Incubation

1. Remove any spawning shelters with eggs and place in dechlorinated water to cover in a 2-3 liter beaker (if fish are in the process of spawning do not remove the shelter until spawning is completed). Label beaker with the date and spawning tank number.
2. Add approximately 1 ml methylene blue per beaker and place in temperature controlled water bath ($25^{\circ} \pm 2^{\circ}\text{C}$). Place active air stone in beaker. Air flow should be fairly high.
3. After 24 hours, water containing methylene blue is replaced with clear dechlorinated water. Beaker and eggs are rinsed with dechlorinated water. Eggs can be 'rolled' off shelters at this point into clear dechlorinated water. Beakers are returned to 25° water bath and air stones (which have also been rinsed) are replaced in beakers.
4. After three days of incubation, air flow is reduced to avoid injury to hatching fry. Air flow is checked daily. Some eggs will progress faster than others.
5. Eggs should hatch after 5 or 6 days at 25°C . If temperature is lower it will take longer. When eggs have hatched, siphon most of the water from the beaker taking care not to catch the fry against the siphon. (Siphoning hose consists of a length of air line hose with a small square of nytex netting attached to one end with a rubber band). Pour fry into rearing dish. Check to see if any fry have stuck to the sides of the beaker. If so, they may be rinsed into the dish with dechlorinated water. Add enough dechlorinated water to the dish to raise the water level to within one inch of the top.
6. Fry from different beakers may be combined as long as hatch dates are within 3-5 days. Dishes should not be overcrowded and may need to have populations divided as fry mature. Each dish receives a batch number which consists of the letters BMI followed by the date of the earliest hatch (Example - BMI091087). A log sheet (Fig 12) should be kept on each dish, with appropriate routine care information plus the spawning tank number from which the eggs originated.

Egg Incubation and Fry Culture Log Sheet

Feeding Regime:

A.M. _____

P.M. _____

Weekends

Tank #:

Batch #: _____

Species: _____

[illegible]

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

C.3 Routine Handling of Fry Culture Dishes

1. Fry less than 90 days old are currently held in "Carolina" dishes in dechlorinated tap water.
2. Culture dishes are maintained with active air stones with gentle aeration at room temperature.
3. Fry are fed live 48 hr. old Artemia twice daily which have been drained of salt water and rinsed with dechlorinated tap water. The amount of food depends on the number of fry per dish and their size. Any excess food is siphoned off two hours after feeding.
4. At least two-thirds (2/3) of the water in each dish plus any accumulated wastes in the bottom of the dish are siphoned off daily and water replaced with clean dechlorinated water. (Siphoning hose consists of a length of airline hose with a small square of nytex netting attached to one end with a small rubber band.)
5. All information concerning care and feeding of fry should be logged daily on the Fry Culture log sheet (Fig. 12).

(2)

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

C.4 Obtaining Fish From an Outside Supplier

1. At least 24 hours (but preferably 1 week) before receiving fish, set up a clean holding tank with appropriate water (fresh, salt), with or without a filter system.
2. Monitor presence of chlorine, temperature and salinity to comply with supplier's recommendations for culturing. Use a chiller or heater, if necessary.
3. Aerate using airstones or filter system.
4. When fish arrive, open the plastic bag and add an active airstone. After water temperature in the bag reaches temperature of the holding tank, place bag containing fish and water into holding tank. Make a small opening in the bag to allow the fish to swim out on their own. (If this is impossible because of the size of the holding tank, gently transfer fish from bag to tank with a clean net.)
5. Enter fish batch number in the fish log file and fill out a Fish Culture log sheet for the new holding tank.
6. Make frequent checks at first to make sure fish are not under stress. (Stress may be indicated by abnormal behavior in swimming and gill movements.) Check temperature.
7. If indicated, a prophylactic treatment using Maracyn may be in order. Follow directions for treatment included with Maracyn.
8. Feed appropriately as directed by supplier or testing guidelines.

APPENDIX XI. *Hyalella azteca*

Contact: Marcia Kelly Nelson
U.S. Fish and Wildlife Service
National Fisheries Contaminant Research Center
Route 2 4200 New Haven Road
Columbia, MD 65201 (314)-675-5399

XI.1 Significance - *Hyalella azteca* (Saussure), Amphipoda, has many desirable characteristics of a test organism: short generation time, easily collected from natural sources or cultured in the laboratory in large numbers, and data on survival, growth, and reproduction can be obtained in toxicity tests (36). Landrum and Scavia (37), Nebeker et al. (22), and Ingersoll and Nelson (4) have successfully used *H. azteca* in sediment toxicity testing and have shown it to be a sensitive indicator of the presence of contaminants associated with sediments. Ingersoll and Nelson (4) report *H. azteca* to have a wide tolerance of sediment grain size. Sediment ranging from >90% silt- and clay-size particles to 100% sand-size particles did not reduce survival or growth in the laboratory.

XI.2 Life History and Life Cycle - The life-cycle of *H. azteca* can be divided into three distinct stages according to Cooper (36): (1) an immature stage, consisting of the first 5 instars; (2) a juvenile stage, including instars 6 and 7; and (3) an adult stage, the 8th instar and older. The potential number of adult instars is large and growth is indeterminate such that old adults can be much larger than younger adults (38). DeMarch (39) indicates that juvenile *H. azteca* can complete a life-cycle in 27 days or longer depending on temperature.

XI.2.1 *H. azteca* is an epibenthic detritivore and will burrow in the sediment surface, and Hargrave (40) has demonstrated in laboratory experiments that *H. azteca* digests bacteria and algae from ingested sediment particles (< 5 μ m), further illustrating sediment interactions by *H. azteca*.

XI.2.2 Sexual dimorphism occurs in *H. azteca*, the adult male is larger than females and has larger second gnathopods (41).

XI.2.3 DeMarch (41) indicates that the number of young produced per adult female is optimum at temperatures of 26-28 °C. Whereas, Cooper (36) and Strong (38) report that maximum brood size is more dependent on the size of a adult amphipods than on temperature.

11.5 Brood Stock - Brood stock can be obtained from another laboratory or commercial source. *H. azteca* brought into the laboratory should be acclimated to the culture water by gradually changing the water in the culture from the water in which they were transported to 100% culture water over a period of 2 or more days. *H. azteca* should be acclimated to the test temperature by changing the water temperature at a rate not to exceed 2°C per 24 h, until the desired temperature is reached (41). Brood stock should be cultured so they are not unnecessarily stressed. To maintain *H. azteca* in good condition and avoid unnecessary stress, crowding and rapid changes in temperature and water quality characteristics should be avoided.

11.6 Handling - *H. azteca* should be handled as little as possible. When handling is necessary, it should be done as gently, carefully, and quickly as possible, so that the amphipods are not unnecessarily stressed. Amphipods should be introduced into solutions beneath the air-water interface (4). Any amphipods that touch dry surfaces, are dropped, or injured during handling should be discarded. Removing animals from sieves may form air bubbles on body surfaces causing animals to float on the water surface. Any "floaters" should be placed into the water column using a probe. If the animals continue to float they should be removed and not used for toxicity testing.

11.7 Age - Tests with *H. azteca* may be started with juvenile organisms, (third instar) about 2-3 mm in length (4,22). To obtain *H. azteca* for testing, amphipods are separated from the leaf material by scooping up the material with clinging amphipods, and placing the leaves on a 5-10 mm mesh screen, which is placed over a collecting pan containing 2 cm of culture water. Culture water is then sprinkled on the leaves while turning and separating the leaves. Mixed age *H. azteca* are washed from the leaves and pass through the screen into a collecting pan (22). To separate the juvenile amphipods from the larger adults a sieve stack (U.S. Standard) #30 (600 µm), #40 (425 µm), and a #60 (250 µm) can be used. Culture water is rinsed through the sieves and juvenile animals retained by the #60 sieve are washed into a collecting pan while the larger animals in the top sieves (#30 and #40) are returned to the culture. The juvenile amphipods are then placed in 1-L beakers containing culture water (about 200 amphipods/beaker) and kept in the dark at the temperature of the culture with gentle aeration. *H. azteca* can be isolated from the culture prior to the start of the sediment toxicity test.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

A.3 Dechlorinator Maintenance

1. Dechlorination is currently being handled by a Culligan Model #446468, dechlorination system. This system employs an activated carbon filter. Backflushing is automatic and occurs every fourth day.
2. Routine maintenance which includes carbon and bacteriological filter changes is handled by a company representative and records are kept of service calls and maintenance schedules by laboratory personnel. Service calls occur approximately every fourth month, or more often if indicated by water quality.
3. All information concerning maintenance is recorded on the Dechlorinated Tap Water and Dechlorinator log sheet (Fig. 3).

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AR302067

Test Conditions for Acute Fathead Minnow (Pimephales promelas)
Toxicity Tests
(Conforming to Peltier and Weber, 1985; EPA/600/4-85/013)

- | | |
|--|---|
| 1. Temperature (°C) | 20°C ± 2° |
| 2. Light quality: | Ambient laboratory illumination |
| 3. Light intensity: | 50-100 foot candles (ft c)
(ambient laboratory levels) |
| 4. Photoperiod: | 16 h light/24 h |
| 5. Size of test vessel: | 500-1000 ml |
| 6. Volume of test solution: | 500-1000 ml |
| 7. Age of fish: | 1-90 days |
| 8. No. of fish/rep. | 10 (Not to exceed 0.8 g fish per
liter of test solution) |
| 9. No. of replicate test vessels
per concentration: | 2 |
| 10. Total No. organisms per
concentration: | 20 |
| 11. Feeding regime: | None |
| 12. Aeration: | None, unless DO concentration
falls below 40% of saturation, at
which time single-bubble aeration
should be started. |
| 13. Dilution water: | BMI dechlorinated water or other
approved water. |
| 14. Test duration: | Definitive test 48 or 96 h
(static or static renewal test) |
| 15. Effect measured: | Mortality - no movement (LC50) |
-

ACUTE BIOASSAY DATA FORM

Initials of Person Setting-up Bioassay

Test Organism; Species: *Pimephales promelas*

Watch:

Ag:

Number of Organisms per Concentration: 20

Test Temperature:
 $20 \pm 2^{\circ}\text{C}$

Dilution Water Used: BHI dechlorinated /

●

Start of Test: Date:

Time:

Test Duration: 96 hour

End of Test: Date:

Time:

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Bioassay Procedure Check Sheet

Test ID #: _____ Initials of person setting-up bioassays: _____
 Sample: _____ Test Organism: _____
 Sample Container: _____ No. of Organisms/Concentration: _____
 Diluent: _____ Test Temperature: _____

- (1) Temperature of effluent: _____ °C
- (2) Temperature of diluent: _____ °C
- (3) Test Containers used: _____
- (4) No. of Concentrations used: _____ Specify: _____
- (5) No. of Replicates per Concentration: _____
- (6) Test containers labeled with concentration, replicate #, and ID # ? _____
- (7) How was diluent delivered to containers? _____
- (8) What glassware was used to measure diluent? _____
- (9) How was effluent delivered to containers? _____
- (10) What glassware was used to measure effluent? _____
- (11) Specify below # mls of diluent and effluent measured out per concentration in this test: _____

Concentration (% effluent)	Diluent	Effluent	Total
1			
2			
3			
4			
5			
6			
7			

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

G.2 Static Acute (48h) Daphnid Toxicity Tests
Ceriodaphnia dubia, Daphnia pulex, Daphnia magna
(Conforming to Peltier and Weber, 1985; EPA/600/4-85/013)

1. The test temperature is $20 \pm 2^{\circ}\text{C}$.
2. All test organisms must be <24 h old at the start of the test. All organisms used in the tests are cultured at BMI. For methods of obtaining test organisms, see SOPs D.2, D.4, and D.6 of this document.
3. The test volume is 100 ml and Pyrex 100 x 50 mm dishes or 50 mls in 60 ml disposable beakers are used as the test chambers.
4. Follow steps 1 - 10 in the General Laboratory Bioassay Procedures (G.1). BMI Acute Bioassay Data Form (Fig. 19) is employed for recording data.
5. Analyze test data using U.S. EPA computer program for analysis of acute toxicity test data.

Test Conditions for Acute Daphnid effluent Toxicity Tests
Ceriodaphnia dubia, Daphnia pulex, Daphnia magna
(Conforming to Peltier and Weber, 1985; EPA/600/4-85/013)

1. Temperature (°C)	20 ± 2°C
2. Light quality:	Ambient laboratory illumination
3. Light intensity:	50-100 foot candles (ft c) ^b (ambient laboratory levels)
4. Photoperiod:	16 h light/24 h
5. Size of test vessel:	300 ml (or 60 ml)
6. Volume of test solution:	100 ml (or 50 ml)
7. Age of test organisms:	1-24 h (neonates)
8. No. animals per test vessel:	10
9. No. of replicate test vessels per concentration:	2
10. Total No. organisms per concentration:	20
11. Feeding regime:	None
12. Aeration:	None, unless DO concentration falls below 40% of saturation, at which time start gentle, single- bubble aeration.
13. Dilution Water:	BMI dechlorinated water or other approved water.
14. Test duration:	Definitive test-48 h (static tests)
15. Effect measured:	Mortality - no movement of body or appendages on gentle prodding (LC50)

^b - ft c = foot candles

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

D.2 Obtaining Daphnia pulex Neonates for Testing

1. This procedure should be implemented 24 hrs prior to the start of the test and will produce enough organisms less than 24 hrs old for one test.
2. Prepare three (3) Carolina dishes with about 1000 mls each dechlorinated tap water and allow them to reach 20° C (room temperature).
3. From a healthy stock culture, carefully transfer 10 - 12 adult females bearing embryos in their brood pouches to each Carolina dish using a wide bore Nalgene pipet. (Avoid exposing organisms to air by releasing under the surface.)
4. Feed each dish 2 ml Daphnia food (SOP F.2) and label dish with date and time of day. Leave at room temperature (20° C).
5. Young found in the dishes the following day are used in test.
6. Avoid using organisms from cultures that are producing ephippia.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

H.2 Fathead Minnow (*Pimephales promelas*)
Larval Survival and Growth Test
(Conforms to Horning and Weber, 1985; EPA/600/4-85/014)

1. Test temperature is $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
2. The test chambers are 125 x 65 mm Pyrex borosilicate glass dishes and the test volume is 500 ml. Information concerning all aspects of testing is recorded on the Fathead Minnow Larval Survival and Growth Test log sheet (Fig. 23).
3. Test organisms must be 1-24 hours old at the start of the test and should be the product of at least three separate spawnings.
4. Six or seven test concentrations, including a control, are used. At least 20 organisms are exposed to each concentration in at least 2 replicate test chambers.
5. Stock solutions (1000 ml) of each test concentration are prepared using the appropriate volumetric glassware. Unless otherwise specified by the client or regulatory agency, BMI dechlorinated/fathead minnow culture water is used as diluent. Prior to the preparation of the stock solutions total residual chlorine is measured in the dilution water. If the TRC is greater than 0.01 mg/L, the water is not used in the test. All stock solution containers must be properly labeled with concentration and test I.D.#.
6. Adjust temperature of stock solutions to $25^{\circ} \pm 2^{\circ}\text{C}$. Measure and record temperature, dissolved oxygen, conductivity, and pH of the control (0%), intermediate, and high concentrations. If the dissolved oxygen concentration of any stock solution is less than 40% saturation, all stock solutions are aerated prior to the start of the test until 40% or greater saturation is reached. Alkalinity and hardness of the control and high concentration are measured.
7. Label test containers with concentration, test I.D.#, and replicate #. Measure and distribute stock solutions to appropriate test chambers.
8. The test is initiated by placing larvae, one or two at a time, into each test chamber in sequential order, until each test chamber contains 10 larvae, for a total of at least 20 larvae for each concentration. Verify and record the # of organisms in each chamber. The amount of water added to the chambers when transferring the larvae to the compartments must be kept to a minimum (<3 ml) to avoid unnecessary dilution of the test concentrations. A disposable polyethylene pipet (bore size 5 mm) is used to transfer the organisms. Randomize the position of test chambers at the beginning of the test.

9. Feed each test chamber 0.1 ml (approximately 700-1000 organisms) of a concentrated suspension of newly hatched (less than 24 hours old) brine shrimp (Artemia) nauplii three times daily at 4 hour intervals. The nauplii are rinsed with distilled water before use.
10. Test solutions are renewed daily using freshly collected samples. First, test chambers are cleaned by removing uneaten and dead brine shrimp and other debris with a siphon hose (dia 3 mm). Care must be taken not to siphon out the larvae. If larvae are removed while cleaning, replace them with a pipet. Continue siphoning until 80-85% of the old test solution has been removed. Replace the volume removed with the freshly prepared solution. The new solution should be added slowly, by pouring down the side of the test chamber to avoid excessive turbulence for the larvae. The necessary water chemistry analyses are performed before and after renewal and recorded as such. The number of live organisms in each chamber is also recorded daily and dead larvae are discarded.
11. The test is terminated after seven days of exposure. At termination, the living larvae in each test chamber are counted and preserved as a group, in 4% formalin, and are dried and weighed at a later date. Immediately prior to the dry weight analysis, the preserved larvae from each test are rinsed in distilled water. The group of rinsed larvae from each test chamber are transferred to a tared weighing boat and dried at 100°C for a minimum of 2 hours. Immediately upon removal from the drying oven, the weighing boats are placed in a dessicator to prevent the absorption of moisture from the air, until weighed. The weights should be measured to the nearest 0.1 mg.
12. Seven day NOEC and LOEC values are calculated using U.S. EPA computer programs for analysis of chronic bioassay data.

Fathead Minnow (Pimephales promelas)- Larval Survival and Growth Test

Experiment I.D. #

[illegible]

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Fig. 23

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BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

D.10 Artemia (Brine Shrimp) Cultures

1. Fill one clean quart (0.946 l) mason jar to within 1/2 inch of the neck with dechlorinated tap water (approximately 840 ml).
2. Add 2 tablespoons of artificial sea salts.
3. Add 2 teaspoons of Artemia eggs.
4. Stir to mix with a stir rod.
5. Aerate vigorously for 48 hours. Spray eggs that stick to the sides of the jar back into the culture with a minimum amount of dechlorinated or distilled water from a sprayer bottle. This is done twice daily.
6. Two jars are maintained in order to have a supply of newly hatched Artemia every 24 hours. Jars are labeled A-1 and A-2 and set up on alternate days. Artemia left over from a 48 hour old culture and not used the same day is poured into a jar marked 'standby' to be used for afternoon feedings.
7. Before Artemia are fed to organisms, jars with airstones removed are allowed to sit in front of a light which is concentrated at the bottom of the jar to allow the Artemia to separate from the cysts.
8. Artemia are removed by carefully pipetting them from the bottom of the jar. They are rinsed through a net using a spray bottle of dechlorinated water, and washed from the net into a beaker using the same.
9. Record data on the Artemia log sheet (Fig. 15).

ARTEMIA CULTURES[illegible]

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

H.5 Daphnid Chronic 21 day Tests

1. Test temperature is $20 \pm 1^{\circ}\text{C}$.
2. The test chambers are 100 ml borosilicate glass beakers and the test volume is 80 ml.
3. Organisms used must be 1-24h old at the start of the test (see SOPs for obtaining young Daphnia for tests D.2 and D.4 of this document).
4. Generally 6 or 7 test concentrations, including a control, are used. At least ten organisms are exposed to each concentration, one per test chamber.
5. Stock solutions of each test concentration are prepared using the appropriate volumetric glassware. Unless otherwise specified by the client or regulatory agency, BMI dechlorinated/ Daphnia pulex culture water (hardness = 60-75 mg/L as CaCO_3) is used as dilution water. Prior to the preparation of the stock solutions total residual chlorine is measured in the dilution of the water. If any chlorine is detected (detection limits = 0.01 mg/L) the water is not used in the test. All stock solution containers must be properly labeled with concentration and test I.D.#.
6. Temperature, dissolved oxygen, conductivity and pH are measured in the control (0%), intermediate, and high concentration stock solutions. If the dissolved oxygen concentration of any stock solution is less than 40% saturation, all stock solutions are aerated prior to the start of the test. Alkalinity and hardness of the control and high concentration are measured.
7. Label test containers with concentration, test I.D.#, and replicate #. Check temperature of each stock solution, measure with appropriate volumetric glassware and distribute stock solutions to appropriate test chambers. Add 0.15 ml Daphnia pulex food to each chamber (see SOP F.2 of this document for preparation of Daphnia pulex food).
8. Using a polyethylene disposable pipet (bore size 3 mm) transfer one organism to the 0% 'A' replicate test chamber. Verify and record the transfer on the BMI Chronic Bioassay Data Sheet (Fig. 2). Special care must be taken while transferring organisms; specifically, the organism must be released below the surface of the test solution and as little culture water

as possible should be transferred with the organism (less than 0.3 ml).

9. Transfer one organism to each 'A' replicate test chamber as described above (one pipet should be used to transfer control animals, only). When all 'A' test chambers have one organism, begin transferring organisms to the 'B' replicates. Continue transferring organisms in this manner until all test chambers contain one organism.
10. Randomly place replicates of each concentration in rows. The position of the rows and replicates within each row is randomized each time the test solutions are renewed.
11. The test solutions are renewed three times per week with freshly collected and mixed samples. Stock solutions are prepared and water chemistry analyses are performed as before (these measurements are recorded as 'after renewal'). Starting with the control (0%), organisms are transferred to clean beakers containing 80 ml of fresh test solution containing 0.15 ml Daphnia pulex food. Labels from the old beakers may be transferred or new labels may be made. The following is recorded for each test chamber at this time: no. living adults, no. living offspring, no. dead offspring, and any comments concerning the organisms' health or behavior. Any accidental deaths, such as spilled beakers, are recorded as such. One randomly selected beaker from the control, intermediate, and high concentrations, is selected for water chemistry analyses (these measurements are recorded as 'before renewal').
12. At the end of the test, final survival and reproduction counts are made. The dry weight (dried at 60°C for 72 to 96h) of each individual first-generation daphnid that is alive at the end of the test is determined to the nearest microgram, except that length (distance from apex of helmet to base of spine) may be determined in place of dry weight if a correlation has been shown between length and dry weight.
13. Twenty-one day NOEC and LOEC values for growth and reproduction are calculated using ANOVA and multiple comparison procedures or other appropriate statistical methods. Both an IBM PC/AT and a mainframe IBM 370 are available for data analysis.

Blossay Data Sheet

Test Temperature: _____

Dilution Water Used:

Start of Test: _____

End of Test: _____

Number indicates living offspring, an x beside a number indicates a dead adult w/ offspring, X indicates a dead adult.

No. Young

No. Adults

Conc.	Day No.	A	B	C	D	E	F	G	H	I	J
	Total										
	Total										
	Total										

BMI/SOP3.0

-118-

W-3

AR302081

BKI/SOP3.0

-118-

~~W-3~~

AR30208

Fig. 25

R302081

General Procedures

Sediment into Test Chambers--The day before the toxicity test is initiated (Day -1) each test sediment and reference sediment is sampled and mixed within its storage container and added to the test chambers. Each test chamber will contain the same amount of sediment, determined by weight (100 g).

The sediment aliquot in each test chamber is settled by smoothing with a utensil constructed of polyethylene. After the sediment is placed in the test chambers, overlying water is added. The overlying water is gently poured along side of the test chamber to prevent resuspension of the sediment.

As a measure of quality control for this study, BMI would run an extra control for each species consisting of replicates containing a known good quality sediment and BMI culture water. This control will serve as a test control in the event that the proposed reference site sediment and/or pore water is either contaminated from unknown sources or the sediment is unsuitable for species survival and growth.

Static Testing--BMI dilution water (400 ml) is added to the test chambers. Glass covers are used to cover the test chambers and overlying water is gently aerated (1 bubble/sec) using a glass Pasteur pipet. To allow any suspended sediments to settle, the test organisms will not be introduced into the test system for 24 hours. During this time, sediment/water treatments will be held at 4°C. Water quality parameters are measured prior to the addition of the test organisms.

Water lost to evaporation or splattering is replaced as needed with temperature acclimated overlying water. The water quality of the overlying water in static sediment toxicity test (water hardness, alkalinity, total dissolved solids, and dissolved oxygen) may be altered by the presence of sediment and/or by the addition of food to the test chamber. These changes in water quality may

influence the availability of contaminants to the test organisms.

Documentation

The following describes the contents of the Final Report:

- (a) Name of test and investigator (s), name and location of BMI, and dates of initiation and termination of test.
- (b) Source of reference and test sediments, method for collection, handling, shipping, storage and disposal of sediment.
- (c) Source of overlying water samples, chemical characteristics, and a description of any pretreatment, and results of any demonstration of the ability of a species to survive, grow and/or reproduce in the water.
- (d) Source, history and age of test organisms, scientific name and how verified; source, history and age of brood stock, culture procedures; and source and date of collection of the test organisms, scientific name, name of person who identified the organisms and the taxonomic key used, age, life stage, means and ranges of weights and lengths, observed diseases or unusual appearance, treatments, holding and acclimation procedures.
- (e) Source and composition of food, concentrations of test material and other contaminants, procedure used to prepare food, feeding methods, frequency and ration.
- (f) Description of the experimental design and test chambers (and compartments), the depth and volume of sediment and overlying water in the chambers, lighting, number of test organisms per treatment, date and time of test initiation, temperature measurements, dissolved oxygen concentration (as % saturation)

and any aeration used prior to initiating a test and during the conduct of a test.

- (g) Methods used for, and results (with standard deviations or confidence limits) of physical and chemical analyses of sediment.
- (h) Definition (s) of the effects used to calculate LC50 or EC50s, biological endpoints for the long-term partial life cycle exposures, and a summary of general observations of other effects.
- (i) A table of data on biological endpoints determined by experimental design for specific test organisms in the control and the treatments on sufficient detail to allow independent statistical analysis.
- (j) Methods used for, and results of, statistical analyses of data.
- (k) Summary of general observations on other effects or symptoms.
- (l) Anything unusual about the test, any deviation from these procedures, and any other relevant information.

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Biological Data--During the conduct of the test, observations are made to assess behavioral responses (i.e. "floaters") and reproductive activities (i.e. amplexus). At the end of the test H. azteca are retrieved from the test chambers for survival counts, observable behavioral responses, and any noticeable reproduction (i.e. amplexus, gravid females, young present) and growth. Ingersoll and Nelson (ASTM, 1989) and our own experience, as well, have indicated that without material above the sediment surface, such as the leaves used in culturing, the H. azteca burrow in the top 1 cm sediment surface or are found swimming in the water column. Many of the surviving amphipods, therefore, can be pipetted from the water column before sieving the sediments. At the end of the test the sediment is screened using a #35 (500 um) U.S. Standard size screen cup first by swirling the overlying water to suspend the upper 1 cm of sediment and pouring that slurry into the cup.

Next, a stack of sieves #25 and #40 U. S. Standard size are used to sieve the bulk sediment on order to collect and count the live animals remaining in the sediment. The H. azteca are rinsed from the screens into collecting pans and pipetted from the rinse water. For quantifying growth, H. azteca body length (± 0.01 mm) is measured from the base of the first antenna to the tip of the third uropod along the curve of the dorsal surface. A sediment toxicity test with H. azteca is unacceptable if the average survival on any negative control chamber is less than 80%.

(2)

Hyalalella azteca

Initiation of a Test--Sediments are homogenized and placed in the test chambers on the day prior to the addition of the test organisms as noted above. Dilution water is added and chambers are then covered and aerated, cold (4°C) overnight before H. azteca are added. The test begins when the juvenile H. azteca (24-48 hrs old) are introduced to the test chambers (Day 0). All test chambers are inspected <2 hours after amphipods are introduced to insure that animals are not trapped in the surface tension of the water. These "floaters" do not survive well and are replaced with healthy animals.

Feeding--Following methods of Ingersoll and Nelson (ASTM Draft Guideline for Hyalalella azteca sediment testing, Draft #3, 1989), Purina' Rabbit Pellets will be used as a food for H. azteca in this study. The pellets are ground and dispersed in deionized water. A Teflon' stir bar and a magnetic stir plate are used to homogeneously resuspend the Purina' Rabbit Pellet when aliquots are removed for feeding. If food collects on the sediment, a fungal and/or bacterial growth may start on the surface of the sediment, in which case feeding is suspended for one or more days. A drop on dissolved oxygen to 40% saturation may indicate that all of the food added in the water is not being consumed such that feeding may be suspended for the amount of time necessary to improve the dissolved oxygen.

In static tests Nebeker et al. (Envir. Tox. Chem. 3:617-630; 1984) suggest a feeding regime twice weekly of 200 mg (0.5 ml dry volume) Purina' Rabbit Pellets mixed in 100 ml distilled water for 100 juvenile H. azteca in a 20-L aquarium. Nelson and Ingersoll (ASTM, 1989) recommend feeding H. azteca three times weekly 14 mg Rabbit Pellets per feeding for 20 young amphipods in a 1-L beaker. The latter feeding regime is best suited for the present study.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.1 Procedure for Determining Alkalinity by Titration

1. Fill burette with N/50 sulfuric acid.
2. Using a graduated cylinder, measure two 100 ml portions of sample and transfer each to a 250 ml Erlenmeyer flask.
3. To each flask containing sample, add 2 drops of methyl purple indicator.
4. Slowly titrate N/50 acid into one of the sample flasks, mixing thoroughly by swirling the liquid.
5. At the first appearance of a difference in color between the two flasks, stop titrating and note the amount of acid added to the flask.
6. To calculate alkalinity, use one of the following formulas:
 - a. If 100 ml of a sample are used: $\text{Alk (mg/L of CaCO}_3\text{)} = \frac{\text{(ml of N/50 added)} (10)}{\text{ml of sample}} (100)$
 - b. If 50 ml of a sample are used: $\text{Alk (mg/L of CaCO}_3\text{)} = \frac{\text{(ml of N/50 added)} (20)}{\text{ml of sample}} (50)$
 - c. A smaller volume may be used yielding a different calculation factor.
7. Performance of reagents is checked monthly (or more often if indicated) using commercially prepared standards, currently ordered from HACH Company. HACH analytical standard for alkalinity is kept on hand in the laboratory. Results of quality checks are recorded and kept on permanent file.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.5 Procedure for Determining Chlorine by Amperometric Titration

1. When not in use, the Amperometric titrator is always stored with the agitator and delivery tube immersed in dechlorinated water to which one drop of pH4 buffer has been added.
2. Before measuring chlorine in a sample, the electrodes of the Amperometric Titrator must be stabilized in the following manner:
 - a. Empty the sample container (200 ml beaker). Then, using a wash bottle with clean dechlorinated (or distilled) water and positioning the container to catch the drainage, rinse the delivery tube, electrode and agitator assemblies.
 - b. Discard the drainage and rinse the beaker.
 - c. Fill beaker with tap water to 200 ml mark.
 - d. Add 1 dropper each pH4 buffer and 5% potassium iodide solution.
 - e. Carefully position beaker on the titrator and turn switch to "total".
 - f. Indicator needle should move upscale to full position and sample should remain on agitator for 10 minutes to allow the electrodes to become partially stabilized.
 - g. If enough sample is available, several "test" titrations should be performed before titrating "for the record".
3. To perform the test titration, rinse the electrode and agitator as in 2a.
4. Using a clean 200 ml beaker, measure 200 ml sample to be tested.
5. Add 1 dropper each pH4 buffer and 5% potassium iodide solution.
6. Carefully place beaker on titrator and turn switch to "total".
7. Fill the titrant pipet by opening the "refill" valve to allow level of titrant to rise to 0 ml mark.
8. Titrate slowly by opening "titrate" valve and adding individual drops of titrant, allowing for mixing between drops. Observe and make a mental note of the level of titrant in the pipet before each addition and the resultant downward deflection of the pointer.
9. The end point is reached when the addition of titrant causes no deflection of the indicator needle.

10. Record the volume of titrant used to reach the end point.
11. Turn switch to "off".
12. Total chlorine in mg/L = ml of titrant.
13. For directions on refilling titrant bag, explanations of interference effects and problem solving, and cleaning electrodes, refer to the Instruction Bulletin for series 17T2000 Amperometric Titrator kept in the laboratory files.
14. Performance of meter and reagents is checked monthly, or more often if indicated, using commercially prepared standards specific for chlorine, currently ordered from HACH Company.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.6 Measuring Conductivity (YSI Conductivity Meter)

1. Conductivity is measured in umho/cm on the YSI Model 33 S-C-T Meter, which also reads temperature and salinity.
2. Calibration of Model 33 is done only at the factory of origin, except in the case of emergency. Emergency calibration is described in the YSI Model 33 Manual (filed under "Conductivity Meter, etc." in the laboratory file drawer), pages 9-11. Emergency calibration is temporary; the meter should be returned to the factory for proper recalibration at the earliest opportunity.
3. The procedure for reading conductivity is:
 - a. Switch mode control to OFF. Use meter screw to adjust needle to 0 conductivity if necessary.
 - b. Plug probe into jack.
 - c. Switch mode to RED LINE. Use RED LINE control to align meter needle with red line if necessary.
 - d. Adjust TEMPERATURE dial to temperature of sample.
 - f. Switch mode to appropriate conductivity scale for on-scale meter reading. Read meter and multiply results according to scale used.
 - g. The probe is stored in and rinsed off (between samples) with distilled water.
 - h. Once a month a cell test should be performed. For directions for cell test and for cleaning procedures see manufacturer's manual kept on file in lab.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.7 Dissolved Oxygen (YSI Model 58 D.O. Meter)

1. The YSI Model 58 D.O. Meter may be used in a vertical, horizontal or tilted position and it may be carried or moved during use without affecting its accuracy or stability of measurement. It is currently being used at BMI with the YSI 5750 BOD Bottle Probe which is not equipped with a stirrer. Agitation of the sample must be provided by other means, such as a magnetic stirrer.
2. The BOD probe is fitted with a membrane which must be in perfect condition to obtain a true reading. For replacing membrane and preparing the probe, see pp. 12-13 of the Instruction Manual for the YSI Model 58 Dissolved Oxygen Meter.
3. Connect the prepared probe to the Probe receptacle and screw the retaining ring finger tight.
4. Set the function switch to zero and adjust the display to read 00.0 with the O₂ ZERO Control.
5. Wait at least 15 minutes for the probe to stabilize. A wait is necessary whenever the meter has been OFF or the probe has been disconnected.
6. For calibration, the air calibration method is currently employed at BMI. Calibrate the meter and the BOD bottle probe with the following steps:
 - a. Set the function switch to the % mode.
 - b. Place the BOD probe in a BOD bottle with about 1" of water to provide a 100% relative humidity calibration environment.
 - c. The probe should be zeroed and calibrated at a temperature as close as possible to the temperature of the sample to be measured.
 - d. Set the function switch to zero and readjust display to read 0.00. Switch back to % air saturation mode.
 - e. When the display reading has stabilized, unlock the O₂ CALIB control locking ring and adjust the display to the CALIB VALUE

indicated in the pressure/altitude chart printed on the back of the instrument (also found as a wall chart posted in the lab). Relock the locking ring to prevent inadvertent changes.

- f. Meter is calibrated daily. Results are recorded on the Meter Calibration Log Sheet (Fig. 10).
- g. Failure of the meter or probe to respond correctly after calibration results in checks:
 - 1. Changing a damaged or wrinkled membrane.
 - 2. Cleaning of probe.
 - 3. Inspecting and replacing the cable or connector, if indicated.

If these steps do not restore specification performance, return the probe to the factory for service.

- 7. To measure the DO of a sample after all of the necessary steps for calibration have been completed, place the probe in the sample.
- 8. Adjust the SALINITY control to the salinity of the sample. (Not required when reading % air saturation).
- 9. Turn the function switch to O₂ ZERO and readjust if necessary.
- 10. Turn the function switch to the desired readout mode and read the dissolved oxygen value in mg/L or in % air saturation when the meter reading has stabilized.
- 11. The above is a description of the method used for measuring DO in naturally occurring water and wastewater. For measuring DO in other substances, and for further information or troubleshooting, refer to the Instruction Manual for the YSI Model 58 Dissolved Oxygen Meter kept in the laboratory files.
- 12. Accuracy of performance is checked against the Winkler Method for determining DO once a month, or more often, as indicated.

Meter and Chemical Calibration Record

BMI/SOP 3.0

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.8 Procedure for Determining Dissolved Oxygen
by Azide Modification of Basic Winkler

1. This method can be used for most sewage, effluent, and stream samples or if the NO_2 concentration $> 50 \text{ mg/L}$.
2. Rinse a 300 ml BOD bottle with the water sample to be analyzed.
3. Fill the BOD bottle by inclining it at such an angle so as to minimize aeration.
4. Stopper the BOD bottle, expelling excess sample.
5. Draw up 2 ml of manganese sulfate ($\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$) and add well below the surface of the liquid.
6. Draw up 2 ml of Alkali-iodide-azide reagent (caution - very caustic) and add well below the surface of the liquid.
7. Stopper carefully to exclude air bubbles and mix by inverting at least 15 times.
8. Allow precipitate to settle leaving approximately 2/3 clear supernate above floc.
9. Shake again and allow to settle.
10. Carefully remove stopper and immediately add 2.0 ml of concentrated sulfuric acid (H_2SO_4) by letting the acid run down the neck of the bottle.
11. Restopper and mix by gentle inversion until floc is dissolved.
12. Pour out 203 ml of solution into a 250 ml beaker.
13. Draw up 10 ml 0.025 sodium thiosulfate into a 10 ml pipet.
14. Add sodium thiosulfate in a dropwise fashion with stirring. Titrate to a pale straw color.
15. Add 1 to 2 ml Aqueous Starch Solution which yields a blue color change.
16. Continue titration with stirring until blue color disappears yielding a clear color.
17. Every 1 ml of 0.025 N sodium thiosulfate titrated equals 1 mg/L dissolved oxygen.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.9 Procedure for Determining Hardness by Titration

1. Fill burette with EDTA solution. (This solution is ordered ready to use and stored at BMI).
2. Using a graduated cylinder measure 50 ml of the sample being tested into a 250 ml Erlenmeyer flask.
3. Add 1 - 2 ml of Hardness Buffer Solution.
4. Add 1 - 2 drops of Eriochrome Black T Indicator.
5. Titrate with EDTA solution slowly, mixing thoroughly by swirling flask.
6. Titrate until solution loses reddish tinge and becomes bluish.
7. To calculate hardness as mg/L of CaCO_3 use the equation:
$$\frac{(\text{ml EDTA added}) (1000)}{\text{ml of sample}} = (20) (\text{ml EDTA})$$
8. A suggested modification is to use 25 ml of sample plus 25 ml distilled water and calculate: $\text{HARDNESS} = (\text{ml EDTA}) (40)$.
9. Accuracy of results and methods and quality of reagents are checked with commercially prepared standards once a month or more often as indicated.

BIOLOGICAL MONITORING, INC.
STANDARD OPERATING PROCEDURES

B.13 Measuring Temperature

1. To measure temperature of a liquid, obtain a centigrade (°C) immersible thermometer.
2. Rinse thermometer with dechlorinated water.
3. Place thermometer in test sample and allow approximately two minutes for stabilization.
4. Read thermometer scale for °C.
5. Rinse thermometer thoroughly after use.

SOP NUMBER: C.II.4

DATE: June 1990

TITLE: Field Sampling Protocol for Fish Tissue Metal Analysis

SCOPE: This operating procedure describes the methods for collecting fish to be analyzed for metal bioaccumulation.

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the field.
- Serve as a means to screen for potential bioaccumulation of metals by fish.

EQUIPMENT:

- Ziplock bags.
- Backpack fishshocker (see SOP C.II.6), seines, and/or fishing pole, dip nets.
- Boat, if necessary, and hip boots or chest waders.

PROCEDURE:

1. Collect 50 to 300 grams of fish for each sample. Use a backpack fishshocker (see SOP C.II.6), or fishing pole to retrieve fish.
2. If fish are sparse or reportedly non-existent, use a backpack fishshocker (refer to SOP C.II.6) to confirm lack of fish or to obtain a sample.
3. If a selection of fish is available, try to obtain a large (older) bottom-dwelling game fish, such as an Ictalurus nebulosus (brown bullhead) or an Ictalurus punctatus (channel catfish).
4. Place each fish sample in a clean ziplock bag.
5. Pack fish on ice in a cooler for immediate overnight transport to analytical lab.

SOP NUMBER: C.II.5

DATE: June 1990

TITLE: Analysis of Trace Metals in Fish

SCOPE: See Attachment No. I

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the laboratory.
- Serve as a means to screen the metals content in fish tissue.

EQUIPMENT: See Attachment, No. VII

PRELIMINARY
TO OPERATION: See Attachment

PROCEDURE: See Attachment

ChemAlysis Incorporated
Standard Operating Procedure
Analysis of Trace Metals in Fish

I. SCOPE AND APPLICATION

- 1.1 This method is used for the determination of the following target analyte 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 in fish tissue.

II. SUMMARY OF METHOD

- 2.1 The fish is prepared for analysis by being chopped into small pieces and homogenized in a blender. Samples are then placed in a closed vessel with nitric acid and allowed to stand overnight before being heated at 70 C for 48 hours.

III. SAFETY PRECAUTIONS

- 3.1 Ordinary safety precautions shall be taken with special care in handling concentrated acid.

IV. SAMPLE SIZE REQUIREMENT AND SAMPLE COLLECTION

- 4.1 A 50 to 300 g. fish is needed.
4.2 Samples must be packed in ice in a cooler upon collection.

V. DETECTION LIMITS AND WORKING LINEAR RANGES

- 5.1 See Appendix A for a table of detection limits, method numbers, and working linear ranges.

VI. INTERFERENCES

- 6.1 Samples will be spiked to determine if there is any interference. Corrective measures will be taken as needed such as dilution and/or standard addition.

VII. APPARATUS AND MATERIALS

- 7.1 Blender, Waring, two-speed, stainless steel blade or tantalum blade, glass container capacity 1000 ml, or equivalent.
7.2 Water bath capable of 70 C.

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- 7.3 BOD bottles thoroughly cleaned with detergent and tap water, rinsed with 1:1 nitric acid, then tap water, and finally deionized water.
- 7.4 Wrist Action Shaker.
- 7.5 Nalgene bottles, 175 ml capacity.

VIII REAGENTS

- 8.1 Concentrated Nitric Acid
- 8.2 Deionized water

IX. SAMPLE HOMOGENIZATION

- 9.1 Select a fish weighing between 50 and 300 grams.
- 9.2 Chop fish into approximately 1 inch pellets.
- 9.3 Place pellets of dry ice into blender and pulverize for 10 seconds. The weight of dry ice should be greater than or equal to the weight of the fish.
- 9.4 Add fish and blend at high speed until sample is homogeneous.
- 9.5 Transfer homogenate to a plastic bag and close with rubber band loosely enough so that carbon dioxide can escape.
- 9.6 Place bag in freezer for at least 16 hours until ready to proceed with the digestion step.

X. SAMPLE DIGESTION

- 10.1 Weigh 2.00 +/- 0.001 grams of homogenate and transfer quantitatively to a clean BOD bottle.
- 10.2 Add 5.0 ml conc. nitric acid. Stopper, and fasten to a wrist action shaker and shake for 60 minutes.
- 10.3 Remove from shaker and allow to stand overnight.
- 10.4 Heat bottle in a water bath for 48 hours at 70 C. Cool.
- 10.5 Transfer digestate quantitatively to a 100 ml volumetric flask and bring to volume with deionized water. The sample is now ready for analysis by atomic absorption.
- 10.6 Note that samples to be analyzed for mercury must be digested according to EPA 245.1 using 0.20 grams of homogenate.

XI. CALIBRATION PROCEDURE

- 11.1 Standards of appropriate concentrations are to be made fresh daily from a 1000 ppm stock solution.
- 11.2 The calibration curve must have a correlation coefficient of 0.995 or greater.

XII. ANALYTICAL PROCEDURE

- 12.1 The following target analyte metals shall be analyzed by

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- direct aspiration: aluminum, barium, beryllium, cadmium, calcium, copper, chromium, cobalt, iron, magnesium, manganese, nickel, potassium, silver, vanadium, and zinc.
- 12.2 The following shall be analyzed by the furnace technique: antimony, arsenic, lead, selenium, and thallium.
 - 12.3 Mercury shall be analyzed by the cold vapor technique.
 - 12.4 Samples will be analytically spiked at an appropriate concentration to determine if there are any interferences.
 - 12.5 An EPA Quality Control sample will be analyzed every ten samples. The QC must be within 10% of the true value to be acceptable.

XIII CALCULATIONS

- 13.1 Using the values from the calibration curve, the concentration of each metal in the fish can be calculated as follows:

$$\text{ug/g (mg/kg)} = \frac{(\text{mg/l from curve}) \times (\text{ml final volume})}{\text{weight of wet sample in g}}$$

XIV. QUALITY CONTROL REQUIREMENTS

- 14.1 The following quality control samples shall be run with every twenty samples:
 - (a). Method Blank: should contain less than the detection limit concentration of each target analyte.
 - (b). Laboratory Control Sample: should be within 20% of the true value spiked of each analyte.
 - (c). Laboratory Sample Duplicate: should be within 20% RPD.

XV. REFERENCES

- 15.1 Procedure adapted from Columbia National Fisheries Research Laboratory, 1986. Thomas W. May. CNFRL, Dept. of Interior, Fish and Wildlife Service, Columbia, Missouri and "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", EPA 600/4-79-020.

APPENDIX A

Parameters, Methods, and Detection Limits

PARAMETER	ANALYSIS METHOD	DETECTION LIMIT (mg/kg)	WORKING LINEAR RANGE (mg/kg)
Aluminum	EPA 202.1	10	10 - 250
Antimony	EPA 204.2	0.5	0.5 - 5.0
Arsenic	EPA 206.2	0.25	0.25 - 3.75
Barium	EPA 208.1	2.5	2.5 - 250
Beryllium	EPA 210.1	0.5	0.5 - 50.0
Cadmium	EPA 213.1	0.25	0.25 - 50.0
Calcium	EPA 215.1	250	250 - 1250
Chromium	EPA 218.1	0.5	0.5 - 50.0
Hexachrome	EPA 218.4	0.5	0.5 - 5.0
Cobalt	EPA 219.1	2.5	2.5 - 50.0
Copper	EPA 220.1	1.25	1.25 - 50.0
Iron	EPA 236.1	1.25	1.25 - 125
Lead	EPA 239.2	0.15	0.15 - 3.75
Magnesium	EPA 242.1	250	250 - 1250
Manganese	EPA 243.1	0.010	0.010 - 100
Mercury	EPA 245.1	0.1	0.1 - 5.0
Nickel	EPA 249.1	2.0	2.0 - 50.0
Potassium	EPA 258.1	250	250 - 1250
Selenium	EPA 270.2	0.25	0.25 - 3.75
Silver	EPA 272.1	0.5	0.5 - 50.0
Sodium	EPA 273.1	250	250 - 1250
Thallium	EPA 279.2	0.5	0.5 - 3.75
Vanadium	EPA 286.1	20	20 - 250
Zinc	EPA 289.1	1.0	1.0 - 50.0

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COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

EPA Sample No.	Lab Sample ID.
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100	100

DATE	DESCRIPTION	AMOUNT	BALANCE
10/1/2017	OPENING BALANCE		100.00
10/2/2017	SALES	50.00	150.00
10/3/2017	SALES	75.00	225.00
10/4/2017	SALES	100.00	325.00
10/5/2017	SALES	125.00	450.00
10/6/2017	SALES	150.00	600.00
10/7/2017	SALES	175.00	775.00
10/8/2017	SALES	200.00	975.00
10/9/2017	SALES	225.00	1200.00
10/10/2017	SALES	250.00	1450.00
10/11/2017	SALES	275.00	1725.00
10/12/2017	SALES	300.00	2025.00
10/13/2017	SALES	325.00	2350.00
10/14/2017	SALES	350.00	2700.00
10/15/2017	SALES	375.00	3075.00
10/16/2017	SALES	400.00	3475.00
10/17/2017	SALES	425.00	3900.00
10/18/2017	SALES	450.00	4350.00
10/19/2017	SALES	475.00	4825.00
10/20/2017	SALES	500.00	5325.00
10/21/2017	SALES	525.00	5850.00
10/22/2017	SALES	550.00	6400.00
10/23/2017	SALES	575.00	6975.00
10/24/2017	SALES	600.00	7575.00
10/25/2017	SALES	625.00	8200.00
10/26/2017	SALES	650.00	8850.00
10/27/2017	SALES	675.00	9525.00
10/28/2017	SALES	700.00	10225.00
10/29/2017	SALES	725.00	10950.00
10/30/2017	SALES	750.00	11700.00
10/31/2017	SALES	775.00	12475.00
11/1/2017	SALES	800.00	13275.00
11/2/2017	SALES	825.00	14100.00
11/3/2017	SALES	850.00	14950.00
11/4/2017	SALES	875.00	15825.00
11/5/2017	SALES	900.00	16725.00
11/6/2017	SALES	925.00	17650.00
11/7/2017	SALES	950.00	18600.00
11/8/2017	SALES	975.00	19575.00
11/9/2017	SALES	1000.00	20575.00
11/10/2017	SALES	1025.00	21600.00
11/11/2017	SALES	1050.00	22650.00
11/12/2017	SALES	1075.00	23725.00
11/13/2017	SALES	1100.00	24825.00
11/14/2017	SALES	1125.00	25950.00
11/15/2017	SALES	1150.00	27100.00
11/16/2017	SALES	1175.00	28275.00
11/17/2017	SALES	1200.00	29475.00
11/18/2017	SALES	1225.00	30700.00
11/19/2017	SALES	1250.00	31950.00
11/20/2017	SALES	1275.00	33225.00
11/21/2017	SALES	1300.00	34525.00
11/22/2017	SALES	1325.00	35850.00
11/23/2017	SALES	1350.00	37200.00
11/24/2017	SALES	1375.00	38575.00
11/25/2017	SALES	1400.00	39975.00
11/26/2017	SALES	1425.00	41400.00
11/27/2017	SALES	1450.00	42850.00
11/28/2017	SALES	1475.00	44325.00
11/29/2017	SALES	1500.00	45825.00
11/30/2017	SALES	1525.00	47350.00
11/31/2017	SALES	1550.00	48900.00
12/1/2017	SALES	1575.00	50475.00
12/2/2017	SALES	1600.00	52075.00
12/3/2017	SALES	1625.00	53700.00
12/4/2017	SALES	1650.00	55350.00
12/5/2017	SALES	1675.00	57025.00
12/6/2017	SALES	1700.00	58725.00
12/7/2017	SALES	1725.00	60450.00
12/8/2017	SALES	1750.00	62200.00
12/9/2017	SALES	1775.00	63975.00
12/10/2017	SALES	1800.00	65775.00
12/11/2017	SALES	1825.00	67600.00
12/12/2017	SALES	1850.00	69450.00
12/13/2017	SALES	1875.00	71325.00
12/14/2017	SALES	1900.00	73225.00
12/15/2017	SALES	1925.00	75150.00
12/16/2017	SALES	1950.00	77100.00
12/17/2017	SALES	1975.00	79075.00
12/18/2017	SALES	2000.00	81075.00
12/19/2017	SALES	2025.00	83100.00
12			

Comments:

Signature: _____ Name: _____
Date: _____ Title: _____

U.S. EPA - CLP

1
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Name: _____

Contract: _____

--

Code: _____

Case No.: _____

SAS No.: _____

SDG No.: _____

Matrix (soil/water): _____

Lab Sample ID: _____

Level (low/med): _____

Date Received: _____

Solids: _____

Concentration Units (ug/L or mg/kg dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: _____

Clarity Before: _____

Texture: _____

Color After: _____

Clarity After: _____

Artifacts: _____

Comments:

U.S. EPA - CLP

2A

INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Initial Calibration Source: _____

Continuing Calibration Source: _____

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					M
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

U.S. EPA - CLP

3
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L)						Prepa- ration Blank	C	K
			1	C	2	C	3	C			
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

U.S. EPA - CLP

5A
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO. _____

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix : _____ Level (low/med): _____

* Solids for Sample: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

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6
DUPLICATES

EPA SAMPLE NO.

--

Name: _____ Contract: _____

Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

x (soil/water): _____ Level (low/med): _____

lids for Sample: _____ ‡ Solids for Duplicate: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Coalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Zinc								
Cyanide								

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7

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
Solid LCS Source: _____
Aqueous LCS Source: _____

Analyte	Aqueous (ug/L)			Solid (mg/kg)				
	True	Found	%R	True	Found	C	Limits	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

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8

- STANDARD ADDITION RESULTS

Name: _____ Contract: _____

Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Concentration Units: ug/L

[illegible]

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13
PREPARATION LOG

me: _____ Contract: _____

File: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

1: _____

[illegible]

FORM XIII - IN

3/90

AR302111

14

Lab Name: _____ Contract: _____
Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____
Instrument ID Number: _____ Method: _____
Start Date: _____ End Date: _____

[illegible]

AR302112

SOP NUMBER: C.II.6

DATE: June 1990

TITLE: Field Use of the Backpack Fishshocking Unit

SCOPE: This operating procedure describes the methods for collecting fish via the use of a backpack fishshocking unit.

OBJECTIVES: The activities covered by this procedure:

- Ensure quality control in the field.
- Serve as a means to collect a random assortment of fish in a timely manner.

EQUIPMENT:

- Watertight hip boots or chest waders
- Electrical linemen gloves of at least a 5,000-volt rating.
- Nets (seine nets of 3.2 or 6.4 mm mesh may also be of use, but are optional).
- Smith-Root backpack fishshocker Model VII, complete with shocking unit and anode and cathode rings.
- Sears Die-Hard 120-volt battery (6"x8"x5"), Model 9603-A3F34-U-J4 (a back-up charged battery is recommended).
- Plastic ziplock bags.
- Ice and cooler.

PROCEDURE:

1. Start at the downstream end of the pond or stream and work upstream. This minimizes turbidity and fish are less likely to be swept downstream.

2. Adjust the output voltage and current of the fishshocker in accordance with the conductivity of the water and the size of the fish sought.

Voltage may be as high as 1,200 volts in lower conductivity water. In high conductivity water, lower voltages are used but currents as high as 60 amps may be needed. Remember that larger fish are more sensitive to electrical currents.

3. While wading upstream, probe the anode into likely fish habitat (e.g., pools with over-hanging vegetation) and use a pulsating current. This is more effective than emitting a constant current. Stunned fish will have an involuntary swimming action to follow the anode, a reflex called galvanotaxis.

4. Once fish are stunned, scoop them up using a dip net. Collect only needed fish. Those that are to be returned after examination should be kept in a container of water that is well-aerated and of the same temperature as that of the original body of water from which they came.

5. See SOP C.II.4 for further fish tissue preparation.

**SAFETY
PRECAUTIONS:**

- Never electrofish alone or if you have a heart ailment.
- Use water-tight hip boots or chest waders and electrical linemen gloves of at least a 5,000-volt rating.
- Avoid contact with both the anode and cathode at the same time.

- Be sure that the generator is ground and that all high voltage cables are run through an electrical conduit or in a heavy-duty rubber-covered cord recommended for use in wet areas.
- Make all electrical connections in watertight junction boxes.
- Use nets with insulated handles.
- Know how to administer first aid treatment for electrical shock.
- Have a qualified technician periodically check electrical circuits.
- Disconnect battery when fishshocker is not in use.

REFERENCE: Smith-Root Model VII Electrofisher Manual

SOP NUMBER: C.II.7

TITLE: Fish Sample Container Volumes, Preservation, and Holding Times

SCOPE: This operating procedure describes the appropriate containers, preservatives, and holding times for fish samples.

OBJECTIVES: The activity covered by this procedure:

- Ensure that sample volumes and preservations are sufficient for analytical services.

EQUIPMENT:

- Ziplock bags
- Shipping containers
- Sample labels.

OPERATION PROCEDURE:

1. Refer to Table C.II.7-1 for minimum sample volume, container type, preservation methods, and holding times for particular parameter analyses.
2. Assemble ziplock bags.
3. Apply sample labels to ziplock bags and label project name.
4. Proceed with sampling.

TABLE C.II.7-1

Fish Sample Container, Volume, Preservation, and Holding Time
Requirements for Soil and Sediment

<u>Parameter</u>	<u>Container</u>	<u>Preservation</u>	<u>Holding Time</u>
Metals	50 - 300 g; ziplock bag	Freeze	180 days

APPENDIX B
Resumes of Dames & Moore Project Personnel

AR302118

ANN MARIE ENRIGHT

Title: Staff Engineer

Expertise: Environmental Engineering
Toxicology

Experience With Firm: Staff Engineer, 1989-Present

- Conduct of a Baseline Risk Assessment (BRA) for two sites at the Savanna Army Depot Activity, Illinois. Identified contaminants of concern, selected relevant exposure pathways, calculated risks, and developed soil cleanup criteria.
- Conduct of a risk assessment for the Rentokil Wood Preservers site in Richmond, Virginia.
- Conduct of an Endangerment Assessment for a site in Spotsylvania County, Virginia. Duties included selecting contaminants of concern, identifying environmental fate and transport of contaminants, and conducting an exposure and quantitative risk evaluation.
- Conduct of contamination assessments for the Defense General Supply Center, Richmond, Virginia, and for Ft. Dix, New Jersey.
- Conduct of a risk assessment of a Gibson-Homans facility in Maryland. Identified contaminants of concern, selected relevant exposure pathways, and calculated risks.
- Development of cleanup criteria for building surfaces, for Dowty Aerospace. Cleanup criteria were based on OSHA/NIOSH air exposure levels and inhalation as the major exposure route.
- Conduct of a contamination assessment for the Joliet Army Ammunition Plant, Illinois. Also identified contaminants of concern and calculated human exposure intakes and potential risks.
- Identification of potential receptors in the vicinity of the former Damascus Plant, Virginia, for Mobay Corporation.

ENRIGHT, ANN MARIE (cont'd)

Past

Experience:

Environmental Engineer/Toxicologist, Versar, Inc.,
1988-1989

- Assessment of residual risk to human health and the environment after implementation of remedial actions at hazardous waste sites. The assessments involved analysis of the fate and transport of contaminants, evaluation of human exposure concentrations via various exposure routes, and characterization of the potential residual risk.
- Conduct of property transfer assessments. Duties included physical site inspection, records review, and research.
- Evaluation of the oxidant dissipation kinetics of a bromine-based disinfectant used by a wastewater treatment facility discharging to a freshwater environment. Responsibilities included supervision of field staff, field sampling and analysis, project scheduling, monitoring of budgets, and preparation of final report.
- Management of a sampling project involving monthly, bimonthly, and rainfall sampling of 12 sites at a major airport. Responsibilities included coordinating all sampling events and laboratory analysis, supervision of staff, and time and budget management.
- Conduct of a study to determine the economic feasibility of operating a plastic recycling facility in the Baltimore-Washington area. State and local agencies, as well as private industries, were contacted to identify required permits, potential suppliers of scrap plastics, and potential locations for the facility.

Environmental Engineer, U.S. Environmental Protection Agency, 1986-1988

- Review of risk assessments for controversial Superfund sites.
- Management of a water pollution control project in Puerto Rico and the Virgin Islands. Duties included identification of pollution problems, development

ENRIGHT, ANN MARIE (cont'd)

of work plans, and evaluation of work plan implementation.

- Review of environmental impact statements and assessments. Reviews focused on the potential effects of proposed projects on human health, water quality, and aquatic systems.
- Review of applications for marine discharge waivers from secondary treatment (301(h) waivers). Reviews focused on the potential effects of sewage discharges on marine ecosystems, analysis of attainment of water quality standards, and analysis of proposed post-waiver monitoring programs.

Graduate Fellowship, New Jersey Institute of Technology, 1984-1986

- Conduct of thesis research on toxic metals in food. Duties included literature search of documents and laboratory analyses using electroanalytical techniques.

Academic
Background:

M.S., Environmental Engineering/Toxicology, New Jersey Institute of Technology, 1986
B.S., Environmental Science, St. John's University, 1984

KENNETH E. FISCHER

Title: Senior Scientist

Expertise: Industrial Hygiene
Health and Safety
Environmental Compliance

Experience
With Firm: Senior Scientist, 1989

- As Southern Region Health and Safety Manager, review of plans, health and safety training, medical surveillance programs, and accident prevention measures for Dames & Moore offices in the southeastern United States. Prepared the Dames & Moore Firmwide Health and Safety Manual.
- Management of asbestos abatement projects for the U.S. Army Corps of Engineers at Dover Air Force Base, Walter Reed Army Medical Center, a former Nike missile site, Ft. McNair, and Cameron Station. The projects include asbestos surveys, specification preparation, air monitoring, and contractor oversight.
- Planning of property transfer audits for residential and industrial clients.

Past
Experience: Manager, Environmental Compliance, Health and Safety, and Facilities, U.S. Environmental Protection Agency (EPA) National Enforcement Investigations Center, 1979-1989

- Investigation of numerous natural gas pipeline facilities for PCB handling practices as senior auditor, for Texas Eastern, Inc.
- Safety audits of EPA facilities nationwide.
- Preparation of EPA facilities Safety Manual.
- Preparation of safety protocols, review of safety plans, management of asbestos removal projects, and direction of training and medical monitoring programs.
- Management of regulated materials and worker and community right-to-know programs.

FISCHER, KENNETH E. (cont'd)

- Management of laboratory and office construction, maintenance, and physical security operations.
- Advisor to 27 EPA nationwide laboratory waste disposal operations.
- Instructor for health, safety, and compliance curricula at the Federal Law Enforcement Training Center, Glynco, Georgia.
- Oversight of asbestos abatement projects, environmental audits, and health and safety inspections.
- Responsibility for packing, manifesting, shipping, and disposal of hundreds of drums of hazardous waste.
- Preparation of safety plans for site responses, laboratory activities, and related training.
- Hazardous waste worker safety (OSHA 29 CFR 1910.120) and worker right-to-know (1910.1200) instructor.
- Participation in setting up national contract and staff to manage waste from former sites of illegal drug manufacturing.
- Consultant to public and private sector on packaging, transportation, and disposal of hazardous waste, including:
 - U.S. Mine Safety and Health Administration
 - U.S. General Services Administration
 - U.S. Air Force
 - U.S. Drug Enforcement Agency
 - Federal Bureau of Investigation
 - U.S. Navy
 - U.S. Department of Agriculture
 - U.S. Food and Drug Administration
 - National Institutes of Health
 - U.S. Geological Survey
 - American Council of Independent Labs
 - ALCOA
 - Litton Data Systems
 - Monsanto

FISCHER, KENNETH E. (cont'd)

Program Analyst, EPA, 1976-1979

- Analysis, evaluation, and coordination of national programs regulating the use of pesticides and toxic substances. Prepared studies for management analysis and value of research output using linear programming.

Resident Engineer, Western Electric, Washington, D.C., Service Center, 1970-1976

- Management of cost reduction program and local industrial hygiene efforts. Reduced emissions from plastics refinishing operation and established new techniques for repair of communications equipment.

Assistant, Vitreous State Laboratory, Catholic University, 1968-1970

- Design of environmental temperature control equipment; responsibility for interface and maintenance of ultrasonic, laser, and temperature control equipment for laboratory experiments.

Chairman, Department of Physical Sciences, Colegio San Ignacio, San Juan, Puerto Rico, 1966-1968

Academic
Background:

M.S., Physics and Biomedical Engineering, Catholic University, 1970
B.A., Physics and Liberal Arts, Fordham University, 1964

Professional
Registration:

Certified Industrial Hygienist
Professional Engineer, Virginia, Colorado
Accredited Asbestos Inspector and Management Planner
(under Asbestos Hazard Emergency Response Act regulations)

Professional
Affiliations:

Member, American Industrial Hygiene Association (AIHA)
Member, American Conference of Governmental Industrial Hygienists (ACGIH)
Member and Chair, Joint Hazardous Wastes Committee, AIHA and ACGIH
Board of Directors, Institute for Environmental Auditing

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FISCHER, KENNETH E. (cont'd)

Honors: Listed in Directory of Occupational Health and Safety Specialists, 1st Edition
Listed in Who's Who in the West, 21st Edition
Listed in Who's Who of Emerging Leaders in America, 2nd Edition

Selected
Publications: Fischer, Kenneth E., "Safety in the Chemical Laboratory--
Certifications for Professional Hazardous Materials
and Waste Management," Journal of Chemical Education
(November 1988).

Fischer, Kenneth E., "Self Audits of Hazardous Waste
Operations in Laboratories," Journal of Chemical
Education (September 1987).

Fischer, Kenneth E., "Contracts to Dispose of Laboratory
Waste," Journal of Chemical Education (April 1985).

Fischer, Kenneth E., "Containment Laboratories: Design
Considerations," Occupational Health and Safety
(November-December 1983).

NORMAN W. GABEL

Title: Senior Chemist

Expertise: Exposure Analysis/Risk Assessment
Environmental Chemistry
Organic Geochemistry
Biochemistry

Experience With Firm: Senior Chemist, 1987-Present

- Conduct of exposure and risk assessment studies at the Lone Star Army Ammunition Plant, Texas; Sunflower Army Ammunition Plant, Kansas; Fort Dix Military Reservation, New Jersey; Military Ocean Terminal at Bayonne, New Jersey; Maui Satellite Tracking Station, Hawaii; and Joliet Army Ammunition Plant, Illinois.
- Preparation of preliminary assessments for several industrial sites in Virginia, Maryland, and Washington, D.C.
- Conduct of a fate and transport study of perchloroethylene contamination in groundwater at a large industrial site in northern Virginia.

Past Experience: Senior Scientist, Versar Inc., 1978-1987

- Management of two major U.S. Environmental Protection Agency (EPA) programs on the evaluation of exposure and risk resulting from the production, use, and release to the environment of potentially hazardous industrial chemicals.
- Preparation of comprehensive reports for EPA on the transport and fate of 108 of the 129 priority pollutants (all but pesticides). These studies covered transport processes, including volatilization, sorption, and bioaccumulation; fate processes such as photolysis, hydrolysis, oxidation, and biogradation/biotransformation; and other processes such as haloform reactions and precipitation (for metals).

GABEL, NORMAN W. (cont'd)

- Analysis of the environmental fate and transport of hexachlorobenzene, halogenated solvents, acrylate esters, phenylenediamine, and polycationic flocculants, for EPA. Designed a scheme for assessing the likelihood of dioxin/dibenzofuran contamination in halogenated industrial compounds.
- Assessment of the environmental persistence of over 200 chemical substances used or manufactured at the Penick Corporation site, Lyndhurst, New Jersey, in anticipation of selling this 45-year-old pharmaceutical and pesticide manufacturing facility.
- Management of an EPA task-order type program to develop methodologies to trace selected pollutants from discharging industries to the specific exposed population.
- Evaluation of human exposure to asbestos, aryl phosphates, chlorobenzenes, DEHP, cresols, pyridine, and formaldehyde.
- Direction of exposure assessment studies for asbestos-contaminated vermiculite, DEHP in nonplasticizer uses, cresols, pyridine, and six halogenated solvents, for EPA.
- Principal investigator for a U.S. Army program on the use of modified clays for decontamination of chemical agents; demonstrated the feasibility of using cationically modified montmorillonites to decontaminate the surfaces of objects that have been assaulted with organophosphorus poisons.
- Consultant to the Commonwealth of Virginia on a quantitative assessment of the biodegradability of Kepone.

Senior Post Doctoral Associate, National Academy of Sciences-National Aeronautical and Space Administration (NASA) Ames Research Center, 1975-1977

- Conduct of research on the potential biological and geochemical impact of microwave radiation in the vicinity of proposed receiving antennae for solar energy transmitted from orbiting satellites.

GABEL, NORMAN W. (cont'd)

- Participation in research related to planetary exploration and chemical evolution; investigation of the chemical transformations that can occur in terrestrial environments during planetary development.

Deputy Director, Laboratory of Chemical Evolution,
University of Maryland, 1973-1975

- Originator of the hypothesis that underscores the primal role of physicochemical excitation processes in the development of prebiological systems and the origin of life. First to propose inorganic polyphosphate complexes as the primordial template for biopolymers.

Research Scientist, Illinois State Psychiatric Institute,
1967-1973

- Investigation of inorganic polyphosphates found in animal tissues. Contributions to this work included studies of the formation of these compounds, development of methods for their analysis, and study of the biochemical reactions in which they are involved.

Research Associate, National Academy of Sciences-NASA
Ames Research Center, 1965-1967

- Demonstration that monosaccharides could have originated on the developing, primordial earth through the concentration and reaction of formaldehyde on aquatic and marine clay minerals.

Post Doctoral Fellow (National Institute of Mental Health)
and Research Associate, Department of Psychiatry, University of Illinois, 1962-1965

- Participation in a study program on psychotomimetic drugs. By correlating pharmacological efficacy with stereochemistry and chemical reactivity, designed and synthesized a chemical that caused a marked state of confusion in test animals. In conjunction with this work, invented a unique, patented pharmacological agent, 7-amino-1,3,5-triazaadamantane, which has antiviral and anti-parkinsonian properties similar to amantadine.

GABEL, NORMAN W. (cont'd)

Academic

Background:

Ph.D., Organic Chemistry, University of Chicago, 1961
M.S., Biochemistry, University of Illinois, 1957
B.S., Chemistry, University of Illinois, 1955

Selected

Publications:

Carpenter, C., G. Schweer, G. Stinnett, and N. W. Gabel,
Exposure Assessment for Hexachlorobenzene (U.S.
Environmental Protection Agency, 1986).

Gabel, N. W., S. F. Robinson, P. T. White, M. Buchanan,
and R. Maxfield, Use of Modified Clays for Decontami-
nation of Chemical Agents, Final Report, ARCSL-CR-
83031 (U.S. Army Armament and Research Command,
1982).

Callahan, M. A., M. W. Slimak, N. W. Gabel, I. P. May,
et al., Water-Related Environmental Fate of 129
Priority Pollutants, Volumes I and II, EPA-440/4-79-
029ab (U.S. Environmental Protection Agency, 1979).

Gabel, N. W., "Chemical Evolution: A Terrestrial Reassess-
ment," Progress in Molecular and Subcellular Biology,
Vol. 5, F. E. Hahn, ed. (Heidelberg-New York:
Springer-Verlag, 1976).

Griffith, E. J., N. W. Gabel, and C. Ponnampuruma,
"Phosphorus: The Key to Life on the Primitive
Earth," Origins of Life, Vol. 8 (1977).

Gabel, N. W., "Could Those Rapidly Exchangeable Phos-
phoproteins Be Polyphosphate-Protein Complexes?"
Perspectives in Biology and Medicine, Vol. 15
(1972).

JEFFREY W. NEJEDLY

Title: Assistant Geologist

Expertise: Geology

Experience
With Firm: Assistant Geologist, 1989-Present

- Conduct of Remedial Investigation of a Superfund site in central Virginia, including groundwater, surface water, and sediment sample collection; data management; contamination assessment; regulatory review; and report preparation.
- Conduct of sampling and data collection for assessment of contaminated groundwater and surface water at a hazardous waste site in Massachusetts.
- Conduct of a groundwater sampling program for a Superfund landfill in New Jersey.
- Conduct of several soil contamination assessments, which included sample collection, data evaluation and assessment, and report preparation.
- Preparation of analyses and review of state and Federal environmental regulations.

Past
Experience:

Environmental Policy and Regulatory Analyst, ENS Resources, Inc., 1988-1989

- Preparation of analyses and reports on environmental regulations and legislation.
- Communication with clients concerning the status of pending environmental issues.
- Conduct of research on various environmental topics.

Research Assistant, University of Nevada, 1987-1988

- Conduct of research for a U.S. Department of Energy (DOE) project evaluating basaltic volcanism in southern Nevada. Duties included geologic mapping, geochemical sampling and preparation, geochemical computer modeling, microscopic analysis, and preparation of quarterly progress reports.

NEJEDLY, JEFFREY W. (cont'd)

Assistant Geologic Technician, Sonoma State University,
1985-1987

- Instruction of undergraduate geology courses.
- Participation in organization and implementation of geologic field trips.
- Organization and maintenance of geologic sample collections.

Chemical Technician, Multitech Laboratories, Inc., 1986

- Performance of environmental testing, including acid digestion of water and soil samples for analysis by atomic absorption spectrophotometry, and wet chemical tests for chlorides and cyanides.

Assistant Geologist, Union Oil Company, 1985

- Management of sample collection for an exploration geothermal drilling project. Preliminary identification of geologic units as drilling progressed.

Academic
Background:

B.S., Geology, Sonoma State University, 1987

KAREN E. D. SEIBERT

Title: Staff Environmental Scientist/Biologist

Expertise: Biology
Toxicology
Environmental/Resource Policy
Aquatic Macroinvertebrate Identification

Experience With Firm: Staff Environmental Scientist, 1987-Present

- Preparation of environmental impact assessments, environmental assessments, and habitat assessments for Federal, military, and commercial installations and facilities.
- Design of biomonitoring studies for Superfund sites and environmental impact assessments; incorporation of biota surveys and water quality analysis.
- Analysis of the toxicological impact of contamination on surface water and groundwater quality.
- Management of a water resources laboratory with facilities and equipment for macroinvertebrate, fish, zooplankton, and phytoplankton identification.
- Analysis of the presence of and impact to endangered, threatened, and rare species in localized areas.
- Conduct of stormwater runoff modeling for the Rock Creek estuary study, Anne Arundel County, Maryland.

Past Experience: Natural Resources Biologist, Maryland Department of the Environment, 1985-1987

- Formulation and implementation of a biomonitoring program to be adopted by all Maryland industries discharging hazardous toxic wastes. Duties included interacting with government and industry officials, investigating industries, and designing studies to analyze watersheds impacted by industrial wastewater. Studies included analysis of sediments and fish and macroinvertebrate popula-

SEIBERT, KAREN E. D. (cont'd)

tions, as well as physical and chemical parameters. Responsible for macroinvertebrate identification, statistical interpretations, and technical reports on findings, many of which were used for litigation.

Natural Resources Biologist, Chesapeake Bay Program,
Maryland Department of the Environment, 1984-1985

- Performance of year-round research on an ocean-going vessel throughout the Maryland portion of the Chesapeake Bay. Analyzed chemical, biological, and physical parameters of bay water. Designed research projects and compiled computer data for reports. Addressed public concerns about the environmental health of the bay.

Environmental Scientist, Greenhorne and O'Mara, 1984

- Participation in a domestic sanitation study of several neighborhoods in Maryland in an effort to gain Federal funds to build municipal sewage treatment systems. Interviewed citizens, performed soil studies, and compiled maps for reports.

Investigator/Reports Writer and Draftsman, Earth
Satellite Corporation, Winter 1981, 1982

- Location, mapping, and analysis of environmental conditions of abandoned coal mines in Pennsylvania that affect the health, welfare, and safety of area inhabitants. Participated in extensive surveys and interviewed county officials, miners, and other local residents.

Independent Research, University of Michigan, Summer
1982

- Conduct of research in limnology of the Great Lakes and toxicology at the University of Michigan Biological Station. Research involved studying the effects of varying percentages of treated sewage waste on a zooplankton community in a lacustrine marsh ecosystem.

SEIBERT, KAREN E. D. (cont'd)

Assistant Researcher/Cartographic Assistant, National Geographic Society, Summer 1979, 1980

- Gathering of original boundary proclamations for maps that illustrated selected forests, reclamation projects, parks, and reserves throughout the United States. Performed preliminary layout for map.

Academic
Background:

B.A., Zoology and Environmental Sciences, Ohio Wesleyan University, 1983
Graduate classes taken in toxicology, limnology, natural resources economics, and environmental impact assessment.

Professional
Affiliations:

Member, North American Benthological Society
Member, National Association of Environmental Professionals

ROBERT L. WILEY

Title: Project Environmental Scientist

Expertise: Wetlands
Architectural Planning

Experience With Firm: Project Environmental Scientist, 1990

- Delineation of wetlands for a 200-acre potential industrial development site in South Carolina.

Past Experience: Landscape Architect, Edward D. Stone, Jr., Planners and Landscape Architects, 1988-1990

- Design of sites, grading, and drainage control. Duties also included site analysis, wetlands delineations, project management, and liaison with regulatory agencies.

Teaching Assistant, Ohio State University, Department of Landscape Architecture, 1986-1988

- Instruction of landscape architecture classes in design, construction, and history.

Environmental Engineer, Price River Coal Company, American Electric Power Company, 1981-1985

- Management (including hazardous waste and water quality) of an environmental assessment and response program for a 42,000-acre mining operation. Responsibilities included site analysis and design (including drainage control systems), reclamation planning, permitting, and regulatory compliance programming. Also supervised an environmental system maintenance division, an emergency response team, and design and construction of an environmental impact mitigation system.

Reclamation Inspection, Division of Reclamation, Ohio Department of Natural Resources, 1977-1980

- Supervision of mine inspectors; evaluation of mining and reclamation plans, regulatory compliance, and

WILEY, ROBERT L. (cont'd)

water quality and drainage control systems. Also conducted special investigations.

Conservationist, Soil Conservation Service, U.S. Department of Agriculture, 1974-1976

- Conduct of farm planning and pond and drainage system design. Participated in land and soil surveying.

Self-Employed, 1973-1974

- Organization and operation of timber management company that specialized in timber stand improvement, which involved thinning, pruning, planting, and selective harvest. Hired and trained six employees.

Forestry Technician, Land Management Division, U.S. Department of the Navy, 1972-1973

- Management of harvest, evaluation of timber stand, mensuration, and continuous collection of forest inventory data.

Aircraft Munitions Specialist/Graphic Illustrator. U.S. Marine Corps, 1969-1973

Academic
Background:

M.A., Landscape Architecture, Ohio State University,
1988
B.S., Environmental Biology, Ohio State University,
1977

Professional
Affiliations:

Member, Society of Wetland Scientists
Member, Association of State Wetland Managers
Member, American Association of Zoological Parks and
Aquariums

Professional
Registration:

Registered Landscape Architect, North Carolina

APPENDIX C
Fish Tissue Analysis
ChemAnalysis, Inc.

AR302137

**CHEMALYSIS, INC.
QUALITY ASSURANCE PLAN**

**9705 N. Washington Blvd.
Laurel, Maryland 20723
(301) 776-8388**

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1.0 INTRODUCTION

This document is the Quality Assurance/Quality Control (QA/QC) plan for environmental analysis projects. This plan documents the QA/QC procedures employed for laboratory services. The plan incorporates all applicable elements of the US EPA laboratory guidelines (40 CFR part 136 and US EPA CLP protocols).

The QA/QC plan describes ChemAllysis' Quality Assurance program, its relationship to the laboratory, and its role in ensuring the reliability, validity, and completeness of laboratory operations and analytical results. The plan also outlines QA/QC procedures used for field activities as they relate to this project.

1.1 Quality Assurance Policy Statement

ChemAllysis considers the establishment of a continuing program to ensure the reliability and accuracy of results to be the fundamental responsibility of laboratory management. Therefore, ChemAllysis has implemented a Quality Assurance/Quality Control program of analytical monitoring, data review and procedure documentation to assure the quality of data.

2.0 LABORATORY ORGANIZATION AND RESPONSIBILITIES

2.1 Introduction

The Environmental Analysis Division at ChemAllysis Inc. was created and developed to service industry and government agencies engaged in environmental activities involving site investigation, hazardous waste management, and remediation projects. The company is organized in such a way that it can provide quality data under rapid time constraints at very competitive prices. A ChemAllysis organizational chart illustrating divisions and lines of responsibility is presented in Figure 2-1. The diagram delineates the independence of the Quality Assurance Unit (QAU) from the laboratory operations and data generating sections. The QAU reports directly to the President. This provides independence from laboratory personnel engaged in analysis, reporting and management.

2.1 QA Director

The Quality Assurance Director has the overall responsibility for the development, implementation and administration of the QA program. This effort is supported by QA Coordinators whose responsibilities are described below.

The QA Director establishes QA requirements for each project in coordination with the managing Laboratory Director. Each study receives regular QA inspections and reviews of the data and reports. An analytical quality control program is conducted to ensure the production of valid data. Additional details about this program are available in ChemAllysis' SOPs. The QA Director supervises implementation of the analytical QA program and interacts with the project staff in determining corrective action procedures.

Additional responsibilities performed by the QA Director include: the preparation of written documents defining QA/QC procedures; review and approval of written procedures prepared by others in the lab; scheduling and performance of quality audits; training employees in QA/QC techniques; overseeing inter-laboratory testing programs; maintaining current knowledge of approved methods and other regulatory requirements; serving as a liaison to EPA and other regulatory agencies, and continually assessing the QA program.

2.3 QA Coordinator

The operations of the analytical QA monitoring program are performed by the analysts, the Project Manager and the QA Coordinator. The QA Coordinator reports suspected problems and makes recommendations to the QA Director, the Laboratory Supervisor and the Project Manager following up on corrective

Approved: Andrew S. Jeger
Andrew S. Jeger



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action. The Laboratory Supervisor is responsible for the overall quality of reported data from the group. Each individual analyst is responsible for following ChemAllysis' SOPs and QA program in order to assure data quality.

The QA Coordinator oversees sample receiving operations and has authority to reject samples if integrity is questionable. The sample acceptance criteria is documented in ChemAllysis' SOPs.

2.4 Communications

The QA Director meets with the Laboratory Supervisor and Project Managers on a regular basis to implement the QA analytical monitoring procedures for all phases of a project. Results of QA activities are submitted by the QA Coordinator to the Project Manager through channels to validate analytical results.

Status reports on each project are prepared by the QA unit on a periodic basis to document the findings of study inspections as well as corrective action recommended. The reports are submitted to the President and copied to the Project Manager, the Laboratory Supervisor, and the Laboratory Director.

Any significant problems found during the course of an inspection are brought to the attention of the Laboratory Supervisor immediately. The Laboratory Supervisor is responsible for taking corrective action. The frequency of inspection and status reports is dependent upon the length and intensity of the project.

2.5 Document Control

All raw data, documentation, study protocols, QA reports, and final project reports are retained according to EPA requirements. The documents are stored under secure conditions by the QA unit. Only authorized personnel have access to these records.

2.6 QA Program Assessments

The status of ChemAllysis' QA program is constantly under evaluation by various certifying agencies, clients, and the laboratory QAU. ChemAllysis participates in several external QA/QC programs sponsored by government and independent organizations.

2.7 Personnel

New employees are required to participate in ChemAllysis' rigorous training and testing program before working on an actual project. Prior to assignment, each employee has the knowledge and training to perform the assigned operation and

procedures. This training (including attendance of workshops, seminars, etc.) is documented on an employee training form and maintained in the Personnel Office files.

All employees are trained in ChemAnalysis' Standard Operating Procedures by a project leader and the Laboratory Supervisor. Appropriate time is given to the new employee to learn the sections of the SOPs that apply to their specific responsibilities. A follow-up session is held with the Laboratory Supervisor to answer any questions and evaluate the new employee's grasp of the procedures.

Technicians are instructed in extraction/preparation procedures by their supervisors. Their performance is evaluated by analyzing the extracts and comparing recoveries (refer to Section 5.3). Any problem identified during this testing process is immediately addressed by the Laboratory Supervisor and the Laboratory Director.

An individual resume is maintained for each professional staff member. Resumes are maintained and updated in both the employees personnel file and in the QA Office. In addition to a resume, the file of each employee contains a signed job description.

3.0 FACILITIES, EQUIPMENT AND SUPPLIES

3.1 Facilities

ChemAnalysis' corporate and laboratory operations are located at 9705 North Washington Boulevard, Laurel, Maryland. The facility occupies 15,000 square feet of office, storage and laboratory space. The laboratory facilities have been designed with emphasis on data quality and safety. Individual rooms have been created to minimize laboratory background, cross-contamination, and to maximize productivity and operating efficiency. All rooms are independently ventilated and temperature controlled. The volatile analysis laboratory, for instance, has a separate air ventilating system and air conditioner unit. Wet laboratories are isolated from instrumentation rooms and each contain sufficient hoods, a sink and sealed flooring. Good housekeeping practices are routinely stressed to ensure quality performance.

Specialty gases are stored in an isolated room with constant ventilation. Solvents and laboratory supplies are stored in a separate storage area equipped with a sealed floor and proper ventilation. Pesticide application is not allowed in any part of the building without prior approval by the Laboratory Director.

Access into the building is allowed only in the front lobby area and through a locked back-door to sample receiving. The building is secured after business hours and a guard is employed for evenings and weekends.

3.2 Equipment

Key equipment has a written Standard Operating Procedure which describes the methods for routine inspection, cleaning, maintenance, testing, calibration and/or standardization. Materials and standards required to perform these operations are specified. Frequency of these types of checks varies depending upon the type of instrument and its usage. The SOP defines a realistic and effective schedule for the activities described above.

The persons responsible for performing calibration, maintenance and cleaning are those persons using the equipment, unless otherwise designated by the Laboratory Supervisor.

Written records are maintained to document all inspection, maintenance, testing calibration and/or standardization procedures. The records include the data (month, day and year), a description of activity and actual findings, the name of the person performing the operation, and a statement as to whether the maintenance operations were routine. This information is kept in a log book specific for each instrument.

In the event of equipment failure or malfunction, analyses in process are stopped or transferred to another instrument. Trouble-shooting activities recommended in the operations manual for that instrument are performed and, if necessary, repairs are made by the manufacturer's service department. The equipment is clearly identified as out of service and not used until the required repairs have been made.

Non-routine repairs performed as a result of equipment malfunction are documented in the log book to show the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Quality Assurance monitors the equipment maintenance and calibration program through inspections of instruments and log books every month. Deviations from established SOPs are communicated to the Laboratory Supervisor who is responsible for taking required corrective action.

3.3 Supplies

Reagents are available in many grades and qualities. The analytical procedure determines the quality required for each test. Purchased reagents, acids, solvents and chemicals are dated upon receipt and when opened. If expiration dates are listed on the supplier's label, the material is not used after that date unless recertified as acceptable. If expiration dating is not provided, trained chemists assign expiration dates based on information provided in texts such as the Merck Index.

All prepared reagents and solvents in the laboratory are labeled to indicate identity, concentrations, storage requirements and expiration date. Other information such as date prepared, solvent used, initials, notebook and page references is added as appropriate. No material is used after expiration unless recertified as acceptable.

Consumable items such as reagents, solvents, glassware, and gases are inventoried and ordered by permanently assigned personnel who are familiar with the laboratory operations and the rate of usage.

A library of current vendor catalogs is maintained so that back-up suppliers are available in the event of backorders.

4.0 SAMPLE RECEIVING, STORAGE AND TRACKING

The following describes procedures for the logging, storage and tracking of samples received for analysis by ChemAnalysis. This computerized program was developed to ensure sample and data integrity throughout the receiving, analysis and reporting process.

4.1 Sample Receiving

- 4.1.1 Package/Container Inspection: Each package received is visually inspected for physical shipment damage, liquid leakage, or other visual problems. Damages are reported to the project leader and client immediately and all inspection activities are documented using a sample receipt check list (Figure 4-1).
- 4.1.2 Chain-of-Custody: The receipt of sample packages is documented by recording the name, date and time on an external chain-of-custody form (Figure 4-2), and, if received, a client chain-of-custody form. Some packages may be delivered with a seal. The condition of the seal (broken or sealed) is recorded on all chain-of-custody forms.
- 4.1.3 Preserved Samples: For water samples that require field preservation, the pH is checked with pH strips and recorded as appropriate or inappropriate. Results are recorded on the sample receipt check list (see Figure 4-1). If the pH is inappropriate for the analysis to be performed, the project leader is notified immediately.
- 4.1.4 Handling Precautions: If samples are suspected to be hazardous or are malodorous, gloves are worn when handling samples. Any bottles which may leak should be placed in zip-lock bags for protection.
- 4.1.5 Client Paperwork: All paperwork received in a sample package is copied and attached to the work-order (Figure 4-3). Original paperwork is to be kept in the sample receiving notebook accompanying the sample tracking information (Figure 4-4).

4.2 Sample Logging

Each set of samples from a single source or client is logged into a dBASE III data file named SETLOG.DBF. Each sample represents one record in the dBASE file. Lab IDs are sequentially numbered with the first two digits referring to the year (Ex: 880001). All sample information associated with the samples is entered into the first record. Information which does not vary from one sample to another is computer copied to all other records to minimize data entry.

- 4.2.1 Labels for the samples (Figure 4-5) are printed containing five lines of sample information derived from the data file and attached to each sample.
- 4.2.2 A hard copy of the sample set logged-in is printed using the data file, attached to original client paperwork and stored in the sample tracking notebook.
- 4.2.3 A laboratory sample chain-of-custody form (Figure 4-6) is printed for each set of samples. This sheet is used for signing in and out samples from sample receiving. Each sheet is stored in a notebook labeled ChemAnalysis Sample Custody Records.
- 4.2.4 A sample work order form is printed and distributed to the project leader of the sample set and to QA/QC. Copies of any client paperwork are attached.

4.3 Sample Storage

Following completion of sample log-in, all samples are to be stored in appropriate areas. Detailed listings describing where samples will be stored and under what conditions are filed in Sample Receiving. Samples are stored according to EPA requirements on containers, storage temperatures and hold times.

Samples are transferred and/or disposed of under the following conditions:

- o Protocol stipulates storage period and/or disposal.
- o Client requests samples shipped back.
- o Client provides written permission to dispose samples.

All transfer/disposal actions are recorded in the Tracking Notebook.

4.4 Sample Tracking

4.4.1 Work-Order: The purpose of the work-order is to alert lab personnel and QA/QC that samples have been received and logged in. It also accompanies the samples to identify which analyses are to be performed. Upon completion of work, the verbal and written report date is listed on the work-order which is then returned to sample receiving. A copy of the form is filed with the raw data and report. Sample receiving will enter the post-analysis data (date reported) into the dBASE file.

4.4.2 Tracking and Sorting Information: The data base file fields can be indexed and sorted for analysis and sample receipt information, and work performed at any point after samples have been logged-in.



SAMPLE RECEIPT CHECK LIST

	YES	NO	N/A	COMMENTS
1. Client paperwork submitted with samples?	_____	_____	_____	_____
2. Chemalysis sample submittal form filled out?	_____	_____	_____	_____
3. Chain-of-Custody forms properly signed?	_____	_____	_____	_____
4. Client paperwork corresponds with samples received?	_____	_____	_____	_____
5. Samples transported accordingly per analyses: a) Frozen	_____	_____	_____	_____
b) Chilled	_____	_____	_____	_____
c) Ambient	_____	_____	_____	_____
6. Sample containers received intact?	_____	_____	_____	_____
7. Sample seals intact?	_____	_____	_____	_____
8. Presence of air bubbles (VOA's)?	_____	_____	_____	_____
9. pH < 2 for Metals?	_____	_____	_____	_____
10. pH > 12 for Cyanide?	_____	_____	_____	_____
11. Project leader notified of any discrepancies?	_____	_____	_____	_____
12. General condition of samples upon receipt:				_____ _____ _____ _____

Figure 4-1

Logged by: _____

Date: _____

CHEM ANALYSIS INCORPORATED

WORK ORDER FILE

COMMENTS

ANALYST MATRIX STORAGE DATE SAMPLED DATE RECEIVED LOG BY

/ / / /

CLIENT ID ANALYSIS

LAB ID

CLIENT (PROJECT NO)

()

Figure 4-3

Work-Order Sheet

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CHEMALYSIS INCORPORATED

SAMPLE TRACKING RECORD

CLIENT (PROJECT NO)	LAB ID	CLIENT ID	ANALYSIS	MATRIX	STORAGE	DATE SAMPLED	DATE RECEIVED	LOG BY	DISPOSAL	DATE	COMMENTS
()						/ /	/ /	/ /	/ /	/ /	/ /

Figure 4-4
Sample Tracking Record

CLIENT:
LAB ID:
CLIENT ID:
DATE SAMPLED: / /
ANALYSIS:

Figure 4-5 Sample Label

CHEMUNYIS INCORPORATED
 LABORATORY CHAIN OF CUSTODY RECORD

(DATE/TIME/ANALYST)

LAB ID	DATE RECEIVED	REC BY	LOG OUT 1	LOG IN 2	LOG OUT 2	LOG IN 3	LOG OUT 3	LOG IN 4	LOG OUT 4	LOG IN 5	DISPOSAL DATE
	/ /										/ /

Figure 4-6
 Laboratory Chain-of-Custody Form

5.0 ANALYTICAL ACTIVITIES

5.1 Standard Operating Procedures

All of the common laboratory activities and methods are outlined in the form of Standard Operating Procedures (SOPs). This ensures uniformity and consistency in analytical testing and general laboratory functions. SOPs are available for routine analytical methodologies as well as daily laboratory activities. The SOPs are reviewed and approved by laboratory supervisors and QAU prior to acceptance as an official document. Following approval the SOPs serve as ChemAllysis accepted protocols for normal activities. A Sponsor's protocol, however, supercedes the respective ChemAllysis protocol.

5.2 Choice of Environmental Methods

Environmental analysis methods have various advantages and limitations pertaining to their sensitivity, specificity and application to a particular matrix. The selection of analytical methods for environmental samples is usually dictated by regulating agencies, requests by clients, or laboratory choice. The choice of analytical methods by the laboratory is primarily based on the following criteria:

- o Chemical Compounds for Identification
- o Matrix Interferences and Separation Capabilities
- o Detection Limit and Range of Quantitation
- o Accuracy and Precision

5.3 Operator Training

Only analysts who have received proper instruction for a particular method, and successfully completed performance test samples, are allowed to perform an analysis. Analysts are trained only by chemists who have demonstrated skills in that particular analysis. A new trainee is required to perform a series of spiked samples to evaluate his or her performance before analysis of real samples. The results are evaluated for precision and accuracy and a copy stored in the personnel file.

5.4 Instrument Calibration Procedures

5.4.1 Gas Chromatograph/Mass Spectrometers (GC/MS): Mass Selective Detectors (MSDs) are tuned using autotune software and manual software programs using an RTE series A computer. Daily autotuning is recommended to monitor hardware performance and to optimize source performance. Autotune

reports are initialed by the operator and stored in a notebook. The repeated failure of the system in meeting autotune requirements can indicate an improperly functioning source or other MSD hardware and necessitate cleaning and/or troubleshooting. Instruments are manually tuned from the autotune file to meet EPA spectral abundance criteria. Instruments are checked for tune stability and if necessary are re-tuned every 12 hours of operation.

MSDs are calibrated using five different concentrations of standards containing all target compounds of interest. The relative response factors of the five levels are evaluated for reproducibility and average response. Once the initial calibration is determined to be acceptable (based on US EPA CLP criteria), its stability is verified every 12 hours with a continuing calibration standard. The response factors of the continuing calibration must meet the EPA requirements before samples can be analyzed. Failing continuing calibration data requires performing an initial calibration procedure to update instrument initial responses. Samples are quantitated using the response factors generated by the continuing calibration standard.

5.4.2 Gas Chromatographs (GCs): Gas chromatographs are calibrated using a minimum of three standard concentrations over a 10x range within the region of detector linearity. Linear regression is used for generating a calibration curve for each analyte. The stability of the instrument response and calibration is checked with standards at varying intervals. Sample quantitation is performed from the calibration curve. A standard is placed at the end of a sequence to bracket all samples analyzed.

5.4.3 Atomic Absorption Spectrophotometers: Both the flame and the graphite furnace atomic absorption units are calibrated daily using a 4-point curve plus blank. A standard is always analyzed at the reporting detection limit. Furnace standards are prepared immediately before analysis. The standard curve generated must have a correlation coefficient of 0.995 or greater before any

samples can be analyzed. The accuracy of the calibration is determined by the use of an independent standard, usually an EPA Quality Control solution. If the independent standard is not within 10% of its stated true value, the instrument is re-calibrated. Continuing calibration verification and blank analysis is checked continually at a frequency of 10%.

5.4.4 IR Spectrophotometer: The IR is calibrated using an analytically prepared solution of a reference oil containing n-hexadecane, isooctane, and chlorobenzene diluted in freon-113. Cells containing freon-113 are employed as a signal background check (reference measurement). Calibration curves are prepared by plotting absorbance versus mg petroleum hydrocarbons per 100 ml solution. Standards are analyzed at a rate of 10% to monitor IR stability. A standard is always analyzed at the end of each sample series.

5.4.5 High Performance Liquid Chromatographs (HPLCs): Liquid chromatograph systems are conditioned with mobile phase for approximately 1-2 hours prior to calibration. Instruments are calibrated for all analytes of interest using a minimum of three initial standards. A calibration curve is generated from the responses of the standards to document detector linearity. Additional standards are analyzed at a frequency of every four samples to compensate for detector drift and fluctuations. Analyte concentrations in samples are determined by comparing the response of the nearest external standard possessing the closest analyte response. A standard is analyzed at the end of a sequence to bracket all analyses.

5.4.6 UV/VIS Spectrophotometer: The UV/VIS spectrophotometer is calibrated using at least five analytical standards plus blank. Samples are measured against a reference blank in absorbance units. A continuing calibration standard is analyzed at the end and at a 10% frequency. An EPA Quality Control solution is employed, if available, to check accuracy. The accuracy must be within 10% or the instrument is re-calibrated or the standards are prepared

again. All curves generated must have a correlation greater than 0.995.

- 5.4.7 Analytical Balances: Analytical balances are calibrated before use using a single calibration weight with a weight near the intended range of use. Balances are calibrated over a range of weights monthly. This calibration is recorded in the Balance Calibration Logbook. A logbook is kept for each balance. Balances are periodically checked by a balance service technician.
- 5.4.8 Thermometers are calibrated once a year using an NBS certified thermometer. The record of calibration is kept by the Laboratory Supervisor.
- 5.4.9 Conductivity of the in-house deionized water supply is measured daily with a conductivity meter and recorded in a bound conductivity logbook. The acceptable range for deionized water is 0.5 to 2.0 microhos/cm. If the reading is out of range, the Laboratory Supervisor is notified.

5.5 Preventive Maintenance

- 5.5.1 GC/MS: The performance of each MSD is monitored daily by using the program "Autotune". The autotune data provides information indicating source cleaning requirements and other valuable diagnostic data. The tune results are stored in notebooks for reference. Both the hardware and software are under service contract by Hewlett-Packard. The contract provides 24-hour response for system related problems and/or service. All maintenance performed on the system is kept on file. All data files and files created during sample acquisition and processing are routinely copied to magnetic tape for storage and back-up. Thus, sample results can be re-created and further processed in the future.
- 5.5.2 GC: All gas chromatographs are connected to gas manifold systems with each gas containing on-line purifiers and scrubbers. The stability of each purifier is monitored by either pressure change or adsorbent color change. Purifiers are replaced as needed and the date of replacement recorded on the

daily if continually used. All detectors are cleaned as per instrument manufacturer specifications except electron capture detectors which are sent to the manufacturer for cleaning. Injection ports are also cleaned routinely to eliminate active sites. Capillary column performance is monitored using column test samples containing analytes of various polarities. Peak shapes and responses are recorded and compared with initial data obtained when the column was new. Capillary columns are replaced when performance is below method acceptability limits. All instrument maintenance including detector/injector cleaning, septum change, column change, etc. is recorded in each instrument log book.

6.0 DATA REDUCTION, VALIDATION AND REPORTING

6.1 Data Reduction

Analytical data generated by the GC/MS computer system are reduced into summary tables using various programs. Data are summarized and tabulated to facilitate validation and expedient re-analysis if necessary.

Single analyte chromatographic data from GCs and HPLCs is electronically transferred from integrators to computer spreadsheets via a personal computer and translation software. Sample identification, run number, and integrated data such as peak areas or height, is automatically compiled using the spreadsheet. Sample concentrations are calculated automatically using the information in the spreadsheet via linear regression equations. For multi-analyte methods, integrators calculate sample concentrations based on external standards following each chromatogram. The resulting concentrations are tabulated in a report format following validation.

Inorganic analytical data are transcribed from the instrument print-outs and laboratory notebooks into spread sheets. Pertinent sample information such as moisture, sample weight and volumes are entered on the spreadsheet and the resulting sample calculations are performed electronically.

6.2 Data Validation

The analytical data generated are reviewed and validated by each analyst for accuracy and reliability following completion of analysis. The spectra and retention time data for target compounds present in GC/MS screens are matched to standard data. False positives are deleted from the files. Compounds identified in the GC screens are examined for retention time relative to external standards. False positives are removed and compounds meeting the retention time window requirements are confirmed using another chromatographic system. Surrogate standards are evaluated for individual sample extraction efficiency. Internal standard responses are examined for instrument stability. Matrix spike and matrix spike duplicates are checked to observe sample effects on recoveries and monitor precision between laboratory replicates. The response of analytes present in samples are checked to verify that they are within the calibrated range of the instrument. If they are above the range they are re-analyzed following appropriate dilution. Method blanks are examined for possible laboratory interferences. Matrix spikes results are evaluated for accuracy based on matrix control chart comparison. Control charts are established at ± 2 standard deviations for warning limits and ± 3 standard deviations for unacceptable limits. Data validation summary forms are used to document the review and release of each group of analyses (see Figures 6-1, 6-2, 6-3 and 6-4).

6.3 Data Reporting

ChemAllysis' standard reporting format lists the following information for each sample:

- o Client ID Number
- o Analytical Method
- o Date Sample was Received
- o Date Analyzed
- o Sample Matrix
- o Detection Limit
- o Concentration/Non-Detection

A full volatile and semi-volatile sample analysis package for HSL compounds that conforms to the sample delivery requirements for EPA CLP can be supplied to include the following items:

- o Semi-Volatile Analysis Data Reports
- o Volatile Analysis Data Reports
- o DFTPP and BFB Tune Reports
- o Initial Calibration Data
- o Continuing Calibration Data
- o Surrogate Recovery Summary Report
- o Internal Area Summary Report
- o Matrix Spike Recoveries
- o Matrix Spike Duplicate Recoveries and % Difference Results
- o Blank Summary Report
- o Total Ion Chromatograms
- o Tentatively Identified Compound Summary Report

All raw data are stored on magnetic tape and can be delivered upon request. All raw data can be imported into a variety of IBM PC (MS/DOS) formats for further processing and/or reporting using the HP Vectra ES/12 computer and file translate software.

DATA VALIDATION SUMMARY FORM FOR VOLATILE ANALYSIS

Sample Set:
Project #:

Date:
Method: EPA 624
(CLP criteria)

Yes	No	Requirement
___	___	* Tuned for EFB every 12 hours during sample analysis
___	___	* Response factors and IRSD for initial calibration acceptable for specific compounds
___	___	* EPA QA ampule analyzed to evaluate accuracy of calibration
___	___	* Standard preparation fully documented to allow tracability of standard source and lot number
___	___	* % Difference for continuing calibration compounds acceptable
___	___	* Frequency of blanks, duplicates, and spikes sufficient for sample set (10%).
___	___	* Blank samples free of target compounds
___	___	* Sample duplicate values agree within 10% of original
___	___	* Surrogate recoveries acceptable for QA/QC samples
___	___	* Surrogate recoveries acceptable for all samples
___	___	* All detectable results reported within calibrated range
___	___	* All non-target compounds in sample tentatively identified
___	___	* Internal standard areas of sample within -50% and +100% of continuing standard internal areas
___	___	* Standards and sample spectra for all compounds reported found in the raw data package

Data reviewed and released by:

Date:

Figure 6-1

DATA VALIDATION SUMMARY FORM FOR SEMIVOLATILE ANALYSIS

Sample Set:
Project #:

Date:
Method: EPA 625
(CLP Criteria)

Yes	No	Requirement
___	___	* Tuned for DFTPP every day during sample analysis
___	___	* Response factors and %RSD for initial calibration acceptable for specific compounds
___	___	* EPA QA ampule analyzed to evaluate accuracy of calibration
___	___	* Standard preparation fully documented to allow tracability of standard source and lot number
___	___	* % Difference for continuing calibration compounds acceptable
___	___	* Frequency of blanks, duplicates, and spikes sufficient for for sample set (10%).
___	___	* Blank samples free of target compounds
___	___	* Duplicate values agree within 10% of original sample values
___	___	* Surrogate recoveries acceptable for QA/QC samples
___	___	* Surrogate recoveries acceptable for all samples
___	___	* All detectable results reported within calibrated range
___	___	* All non-target compounds in sample tentatively identified
___	___	* Internal standard areas of sample within -50% and +100% of continuing standard internal areas
___	___	* Standards and sample spectra for all compounds reported found in the raw data package

Data reviewed and released by:

Date:

Figure 6-2

DATA VALIDATION SUMMARY FORM FOR PESTICIDE ANALYSIS

Sample Set:
Project #:

Date:
Method: EPA 608

Yes	No	Requirement
—	—	* EPA QA ampule analyzed to evaluate accuracy of pesticide stock and serial dilutions
—	—	* Standard preparation fully documented to allow tracability of standard source and lot number
—	—	* Standard analyzed at end of sequence to monitor stability
—	—	* Frequency of blanks, duplicates, and spikes sufficient for sample set (10%).
—	—	* Blank samples free of target compounds
—	—	* Sample duplicate values agree within 10% of original
—	—	* Surrogate recoveries acceptable for QA/QC samples
—	—	* Surrogate recoveries acceptable for all samples
—	—	* All detectable results reported within calibrated range
—	—	* Positive responses confirmed on a second column
—	—	* Dual column analysis results exhibiting > 50 ppb of any pesticide re-analyzed by GC/MS

Data reviewed and released by:

Date:

Figure 6-3

DATA VALIDATION SUMMARY FORM FOR INORGANIC ANALYSES

Sample Set:
Project #:

Date:
Method(s): Metals -200 series
Cyanide-325.2
Phenol- 420.1

Yes	No	Requirement
___	___	* Preparation blanks below detection limit
___	___	* Duplicates (Metals only) %RSD acceptable
___	___	* Matrix spike recoveries acceptable
___	___	* Matrix spike duplicates (cyanide) %RSD acceptable
___	___	* Lab control sample recovery 80-120%
___	___	* Standard preparations documented and traceable to manufacturer's lot number
___	___	* EPA QC solution analyzed to evaluate calibration accuracy
___	___	* Continuing calibration performed at 10% frequency with acceptable results
___	___	* Furnace analytical spike recoveries indicate absence of matrix interferences OR appropriate action taken and documented
___	___	* All detectable results reported within calibrated range
___	___	* Raw data, QC cover sheets, digestion and distillation records are present for each parameter

Data reviewed and released by:

Date:

Figure 6-4

7.0 QUALITY CONTROL EVALUATION

7.1 Sample Storage and Holding Times

- 7.1.1 Semi-Volatiles: Water samples must be extracted within seven days of sampling. Extracts are stable in amber glass vials for 40 days at -20°C. Water samples are stored at 4°C protected from light.
- 7.1.2 Volatiles: Water samples must be analyzed within 14 days of sampling. Water samples are stored at 4°C.
- 7.1.3 Pesticides and PCBs: Water samples must be extracted within seven days of sampling.
- Extracts are stable in amber glass vials for 40 days at -20°C. Water samples are stored at 4°C protected from light.
- 7.1.4 Metals: Water samples are stable for all metals except mercury and hexavalent chromium for one to six months. Hexavalent chromium must be prepared for analysis within 24-hours of collection. Samples for mercury must be analyzed within 28 days of sampling.
- 7.1.5 Cyanide: Water samples must be distilled and analyzed within 14 days of sampling. Samples and distillates are stored at 4°C in amber bottles.

7.2 Internal Quality Control Check Samples

QA/QC data are evaluated for each sample and each sample set to monitor analysis and system performance. Data is summarized and stored in the client raw data file as retrievable records. QA/QC data is reported as a deliverable in summary forms.

- 7.2.1 Surrogate Standards: Surrogate standards are added to each sample to monitor individual extraction and analysis performance. Semi-volatile samples must be reported following corrective action (s) if recovery of any one surrogate is below 10%, and, if any two surrogates in any fraction are outside recovery limits. Both analysis results are reported if repeat analysis surrogates are outside the acceptable limits. Volatile samples must be repeated

if any one or more surrogates are outside acceptable ranges. Acceptance criteria are adopted from EPA CLP Statement of Work (Rev 2/88).

7.2.2 Matrix Spike/Matrix Spike Duplicates: Specific compounds are added to samples at a rate of one per ten, or one per batch (whichever comes first) to monitor matrix effects on recovery efficiency and reproducibility. Acceptance criteria are adopted EPA CLP Statement of Work (Rev 2/88). Matrix spike duplicates variance must be within the acceptable values shown above to ensure method precision and reproducibility.

7.2.3 Reagent Blank: Reagent blanks are prepared at a rate of one per set of samples (< 10), one per ten samples, or one per day, whichever comes first. Blank samples producing levels of target compounds greater than detection limits must be repeated and all corresponding samples held in question until corrective measures are taken. Some parameters are allowed five times the method detection limit.

7.2.4 Internal Standard: Areas of internal standards must be evaluated for reproducibility for all runs. Areas that exceed 100% or are below 50% of the areas of the continuing standard are to be considered invalid and repeated.

In addition to the above, Quality Control ampules are obtained from EPA, Cincinnati, Ohio and analyzed frequently to document accuracy of calibrations and methods. Results of these samples are kept on file and made available to clients upon request.

7.3 External Performance Evaluations

ChemAnalysis participates in both drinking water (WS) and water pollution (WP) performance studies in conjunction with State Governments and EPA programs. Results of these studies are used for state certifications and EPA inter-laboratory participation programs.

7.4 Control Charts

The laboratory maintains control charts based on the spike recovery results for analytes in both blank samples and actual water and soil samples. These charts are updated periodically to serve as both a warning indicator and to alert analysts of

out-of-control situations. Warning limits are set at +/- 2 standard deviations and unacceptable limits are established at +/- 3 standard deviations.

7.5 Performance and System Audits

In addition to the Quality Control elements previously discussed, performance and system audits are conducted to monitor the accuracy or bias of the analytical systems. The objective of a performance audit is to:

- o Identify inaccuracies or bias in methods.
- o Estimate precision of results.
- o Detect errors in training procedures.
- o Document overall quality of laboratory performance.

System audits review the entire analytical support systems including data handling, reporting, security, logging, tracking, etc. All of these elements can provide sources of error. System audits are performed periodically by the QA Director.

7.6 Reports to Management

The results of the above audits are made available to all technical managers and the President in the form of reports or memoranda. The reports address problems encountered during the audit and include recommended solutions. The audit reports are filed for future reference.

7.7 Corrective Action Procedures

Corrective action procedures are necessary to eliminate problems or observed failures present in QA audit reports. The procedures used by ChemAnalysis are as follows:

- o Define the problem.
- o Identify corrective course of action plan.
- o Assign responsibility for action plan.
- o Verify elimination of problem.
- o Take measures to avoid future re-occurrences of the problem.
- o Document corrective action.

8.0 RECORDKEEPING AND DOCUMENTATION CONTROL

8.1 Retention of Records

Original data records including notebooks, raw data and instrument print-outs are retained by the laboratory for future review. All data pertinent to a specific project are filed by the project number and archived in a secure on-site storage room for 1 year. Clients are then contacted after the archival period to obtain further instructions about the maintenance of these records.

8.2 Sample Tracking and Disposition

All sample tracking sheets are stored in chronological notebooks and record information concerning the location and disposal dates of all samples. The notebooks are stored in sample receiving and forwarded to QAU for archiving every six months. Environmental samples are stored at proper holding and temperature requirements for the following time periods:

<u>Sample</u>	<u>Duration</u>
Volatiles	3 months
Semi-Volatiles	3 months
Pesticides/PCBs	3 months
Metals	9 months
Inorganics	3 months

Date and means of disposal are recorded on the sample tracking sheet and chain-of-custody sheet.

8.3 Archivable Items

The following items are archived in a secured room accessible only by QAU:

- o Project files
- o Notebooks and log books
- o Chromatograms and mass spectra
- o Raw data and instrument print-outs
- o dBASE files contained on floppy disks
- o GC/MS archive tapes containing data files of all runs

CHEMALYSIS, INC.
STATEMENT OF QUALIFICATIONS

JANUARY 1990

9705 N. WASHINGTON BLVD.
LAUREL, MARYLAND 20723

AR302172

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CORPORATE OVERVIEW

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CORPORATE OVERVIEW

ChemAllysis Incorporated is a state-of-the-art laboratory founded on the commitment to provide analytical services of the highest caliber in the areas of site investigations/remediation, water quality analysis, and pesticide residue studies.

Originally a division of Tegeris Laboratories Inc., ChemAllysis can trace its roots to 1969, when the parent company was founded as Pharmacopathics Research Laboratories. After the acquisition of Borrison Laboratories in 1984, Tegeris earned a national reputation as a highly respected contract toxicology laboratory. In 1988, the decision was made to phase out the animal toxicology studies in order to focus all of the Corporation's efforts on analytical chemistry. Thus, ChemAllysis was created out of Tegeris Laboratories, Inc. in order to better serve the needs of agricultural chemical manufacturers, environmental engineers, industrial facilities, and others requiring high quality, rapid response analytical laboratory services.

Recognizing that the two most crucial elements to a laboratory's success are its personnel and instrumentation, ChemAllysis has assembled a highly qualified staff and acquired the very latest, most advanced analytical equipment available. These scientists bring years of experience conducting thousands of environmental tests for industrial and government clients. Their commitment to providing accurate, responsive analytical services will ensure that ChemAllysis will soon be one of the Nation's leading laboratories.

ANALYTICAL SERVICES

AR302176

ANALYTICAL SERVICES

ChemAnalysis utilizes state-of-the-art instrumentation to perform a wide range of environmental analyses. Services are currently provided in two major areas: environmental monitoring and pesticide/drug registration support.

I. PESTICIDE/DRUG RESIDUE ANALYSIS

ChemAnalysis has formed an experienced team of chemists to assist pesticide manufacturers in the registration of their new products with the EPA and FDA. Each study is assigned a single project leader who has in-depth knowledge of the Federal guidelines and extensive experience in the requirements for registration.

- o Method Development and Validation
- o Pesticide Residue Studies in Plant, Soil, Tissue
- o Drug Residue Studies in Tissues, Plasma and Other Matrices
- o Multi Residue Studies
- o Freezer Stability Studies
- o Environmental Fate Studies - Degradation, Metabolism, Mobility, Dissipation, Accumulation

Both Gas Chromatography (GC) and High Pressure Liquid Chromatography (HPLC) methods are used for residue studies, depending on the protocol. The HPLC detectors include variable wavelength, UV fluorescence and electro-chemical. All registration studies are conducted according to TOSCA and FIFRA protocols. Adherence to GLPs is strictly maintained.

II. ENVIRONMENTAL MONITORING

Whether to investigate possible contamination prior to property transfers, to fulfill permit testing requirements, or to assist in a full scale clean-up, ChemAnalysis has the instrumentation and personnel to perform all the necessary analyses on soil, water, air, sludge, or sediment. Careful scheduling and high-capacity equipment ensure rapid turnaround of results without sacrificing precision and accuracy.

- o EPA Priority Pollutants
- o EPA Hazardous Substances List (from Contract Laboratory Program)
- o Volatile Organics
 - Drinking Water - EPA 502, 503, 524
 - Waste/Ground Water - EPA 601, 602, 624
 - Soil/Solid Wastes - EPA 8010, 8020, 8030, 8240
- o Acid/Base/Neutral Extractables
 - Drinking Water - EPA 525
 - Waste/Ground Water - EPA 625
 - Soil/Solid Waste - EPA 8270
- o Pesticides
 - Drinking Water - EPA 505, 515
 - Waste/Ground Water - EPA 608, 615, 680
 - Soil/Solid Waste - EPA 8080
- o Polychlorinated Biphenyls (PCBs)
 - Waste/Ground Water - EPA 608, 680
 - Oil, Soil, Cement - Proprietary Methods
- o Total Petroleum Hydrocarbons
- o Water Quality (inorganics, organics)
- o Trace Metals - Total, Dissolved, EP Toxicity
- o Poly Nuclear Aromatics (PNAs)
- o RCRA Testing
- o Solvent Screens
- o Identification/Confirmation by GC/MS

Analyses are performed to CERCLA (Superfund), RCRA, or NPDES requirements. Several different reporting formats including CLP are readily available.

III. NUTRIENT ANALYSIS

With the growing interest in nutrition in the public's food supply, ChemAnalysis is expanding its analytical capabilities into nutrient analyses.

Our laboratory with its comprehensive instrumentation and extensive analytical background can offer reliable analyses with short turnaround times.

- o Protein
- o Fat
- o Carbohydrates - Calculated by difference or individual
- o Cholesterol
- o Fatty Acids
- o Vitamins
- o Minerals
- o Calories - Calculated

CERTIFICATION

AR302180

CERTIFICATION

Chemalysis is certified for various organic and inorganic chemical analyses in the following states:

Maryland

Virginia

New Jersey

Massachusetts

District of Columbia

Pending: Florida

FACILITIES

AR302182

FACILITIES

ChemAnalysis occupies a 15,000 square foot facility in Laurel, Maryland, midway between Washington, D.C. and Baltimore, 15 minutes from BWI airport. This central location affords easy access to U.S. Route 95 and allows the Company to effectively service the entire Mid-Atlantic region.

To increase productivity and eliminate the possibility of cross-contamination, the laboratory space is divided into a sample receipt/storage area, wet chemistry areas, and analytical instrument rooms.

Sample Receipt/Storage Area

The limited access sample receipt/storage area includes extensive ambient, cold, and freezer storage space. Separate storage facilities are maintained for volatile organic samples.

Wet Chemistry Area

Samples are prepared for instrumental analysis in a 3 room, 1500 square foot wet chemistry laboratory. Each room is sufficiently self-contained in order that the movement between different areas be kept at a minimum during the course of an analysis. Extensive cabinet space allows the storage of all necessary glassware and equipment near the work areas. In addition, each laboratory contains ample solvent storage space and cold storage facilities for standards. Ten 4-foot hoods ensure a safe working environment.

Analytical Instrument Rooms

Six rooms are designated for specific instrumental analysis:

1. Atomic Absorbption Room for trace metals analysis
2. GC and GC/MS Room for PCB's, pesticides, and extractable organics analysis
3. GC Room for volatile organics analysis
4. GC/MS Room for volatile organics analysis
5. GC Room for pesticide residue analysis
6. HPLC Room for pesticide residue analysis

Other instruments are located in appropriate locations throughout the laboratory.

INSTRUMENTATION

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INSTRUMENTATION

Gas Chromatography/Mass Spectrometry (GC/MS)

- o Hewlett Packard 5970; GC/MS RTE-A Data System; Tekmar LSC-2000 Purge and Trap Concentrator; ALS-2016 Auto Sampler; Wiley NBS/EPA Spectral Library
- o Hewlett Packard 5970; GC/MS RTE-A Data System; Hewlett Packard 7673A Auto Injector; Wiley NBS/EPA Compound Library
- o 1 Hewlett Packard 150 Monochrome Graphics Terminal
- o Hewlett Packard/Vectra ES/12 Model 22 Advance Link Terminal; floppy deliverables
- o 2 Hewlett Packard 2235A Rugged Writer Printers
- o Hewlett Packard 7936 Disk Drive with 306 Megabytes of storage; Hewlett Packard 9144 Magnetic Tape Storage Device

Gas Chromatography (GC)

- o Ten Hewlett Packard 5890 GCs each configured with two or three of the following detectors and an HP 7673 Liquid Auto Sampler and/or a Tekmar Purge and Trap.
 - 4 Electron Capture Detectors
 - 3 Nitrogen-Phosphorus Detectors
 - 2 Flame Photometric Detectors
 - 4 Flame-Ionization Detectors
 - 3 Tracor (Model 1000) Hall Detectors
 - 3 Tekmar LSC 2000
 - 3 Tekmar ALS 2016
- o Packed and Capillary Injection Ports
- o Hewlett Packard 5830A GC; Hewlett Packard 7670 Auto Sampler, Electron Capture and Flame Ionization Detectors; packed column
- o Perkin-Elmer Sigma 2 GC; Electron Capture and Flame Ionization Detector; packed column
- o Shimadzu 9A-GC; Shimadzu A O C-9 Auto Sampler; Nitrogen-Phosphorus and Flame Photometric Detectors; packed and capillary columns

- o Tracor 560 GC; Tracor 770 Auto Sampler; Electron Capture and Flame Photometric Detectors; packed columns

High Pressure Liquid Chromatography (HPLC)

- o Tracor 950 Pump; Tracor 980A Solvent Programmer; tracor 970A Variable Wavelength UV Detector
- o Tracor 950 Pump; Tracor 980A Solvent Programmer; Tracor 970A Variable Wavelength UV Detector
- o 2 Waters 510 Pumps; Waters Automated Gradient Control; Waters 710B WISP Autosampler; Waters 484 Variable Wavelength UV Detector
- o 2 Waters 510 Pumps; Waters Automated Gradient Control; Waters 712 WISP Autosampler; Kratos Spectroflow 783 Variable Wavelength UV Detector
- o Waters 712 WISP Autosampler; Waters 484 Variable Wavelength UV Detector
- o Shimadzu LC-5A Pump
- o Kratos FS950 Fluorescence Detector
- o Gilson Variable Wavelength UV Detector; Gilson 212 Fraction Collector
- o Spectra Physics Winner Auto Lab Data System; Epson Equity 1 + Terminal; Epson EX800 Printer/Plotter
- o ESA Coulochem Model 5100A Electrochemical Detector

Atomic Absorption

- o Varian Spectra 400P Flame, Automatic multi-element
- o Varian Spectra 400 Furnace, Automatic multi-element; Zeeman background correction
- o VGA-76 Automatic Vapor Generation Accessory package
- o 2 IBM PS-2 model 30 computers

Miscellaneous Instrumentation

- o Perkin-Elmer Infrared Spectrophotometer
- o Perkin-Elmer Lambda 3 UV Visible Spectrophotometer

- o Packard TriCarb 300 Scintillation Counter
- o Harvey Biological Tissue Oxidizer
- o Mettler AC 100 Balance
- o Mettler H-10 Analytical Balance
- o Cahn 24 Electro Balance
- o Several IBM PCs and PC compatibles with Hewlett Packard Laser Jet II Printers

DATA SYSTEMS

AR302188

DATA SYSTEMS

Laboratory automation implemented with state-of-the-art technology

A comprehensive data acquisition and management system is responsible for logging, tracking and reporting of samples.

Dedicated PC hosts facilitate direct on-line data capturing with GCs and LCs.

Laboratory instrumentation integrated with PCs provide capabilities for overnight continuous operation, readily accommodating customization facilities for software implementing high-speed data extraction, validation and exporting.

Standardized spreadsheet technology with pre-established templates.

Dial-in modem capability for remote data transfers.

Computer Equipment

- o Hewlett Packard 7936 Disk Drive with 306 Megabytes of storage; Hewlett Packard 9144 Magnetic Tape Storage Device
- o Spectro Physics Winner Auto Lab Data System; Epson Equity 1 + Terminal; Epson EX800 Printer/Plotter
- o Varian DS-654 Multi-Tasking Chromatographic Workstation for acquisition and processing of four signals concurrently
- o 8 IBM Personal Computers
- o 7 Leading Edge DC-2010E Personal Computers (IBM compatible)
- o 2 IBM Personal System II Computers
- o 2 Hyundai Super 286E Personal Computers

QA/QC SUMMARY

AR302190

QUALITY ASSURANCE/QUALITY CONTROL

ChemAllysis considers the establishment of a continuing program to ensure the reliability and validity of results to be the fundamental responsibility of laboratory management. Therefore, ChemAllysis has instituted a Quality Assurance/Quality Control program of analytical monitoring, data review and procedure documentation to assure the quality of data. This comprehensive program, based on the EPA's Contract Laboratory Program and Good Laboratory Practices regulations, is supported by ChemAllysis' Standard Operating Procedures (SOP) and implemented by the Quality Assurance Unit (QAU).

The QAU monitors all phases of each project, from sample receipt and preparation through analysis, data reduction, and report writing to ensure the validity of results and adherence to appropriate regulations.

The major points of ChemAllysis' standard QA/QC Program entail:

- o Extensive training on EPA QA requirements for all incoming laboratory employees
- o Regular inspection of facilities and instrumentation
- o Frequent review and updating of Standard Operating Procedures (SOPs)
- o Comprehensive preventive maintenance program on equipment
- o Careful monitoring of on-going projects to detect any deviations from approved methods or protocols
- o Strict chain of custody procedures
- o Meticulous internal QA/QC practices involving blanks, duplicates, spikes, and matrix samples
- o Thorough data review and validation for all projects

The absolute unbiased precision of an analytical method is confirmed by the periodic analysis of blind duplicates. These samples are analyzed and calculated by the analyst in a normal manner. Corrective measures are implemented immediately if inaccuracies are detected.

ChemAllysis' own QA procedures are periodically tested by analyzing external reference samples (NIOSH, NBS, EPA) whenever available. A record is kept of the laboratory's performance on these evaluations.

REPRESENTATIVE PROJECTS

AR302192

REPRESENTATIVE PROJECTS

In the short time ChemAllysis has been operating as an independent company, it has won the respect of a diverse group of corporations. In fact, ChemAllysis has already been awarded a number of multi-year projects from several of the largest producers in the chemicals industry. Environmental engineers, law firms, and real estate developers have also discovered the advantages of ChemAllysis' service.

Major Clients

American Cyanamid Company
Apex Environmental, Inc.
Cadbury & Schweppes
Ciba-Geigy Corporation
Dames and Moore
Environmental Resources Management, Inc.
Geraghty & Miller
Great Atlantic & Pacific Tea Co., Inc.
Greenhorne and O'Mara, Inc.
Groundwater Technology
Hoechst-Roussel Agri Vet Company
Honeywell, Inc.
IBM
Maryland Department of the Environment
Monsanto Agricultural Company
Pennwalt Corporation
Prince George's County, State of Maryland
Rhône Poulenc Ag Company
Tenerra Engineering Inc.
U.S. Army Corps of Engineers
Virginia Commonwealth Water Control Board
Westinghouse Corporation

ChemAllysis provides analytical services to support site investigation/remediation activities, water quality monitoring, and pesticide registration studies. With a fully equipped laboratory and highly qualified staff, ChemAllysis performs complex analyses for a number of diverse organizations. Below are examples of several past and current projects that illustrate ChemAllysis' wide analytical capabilities.

I. PESTICIDE RESIDUE STUDIES

Numerous Agricultural Chemical Manufacturers

ChemAllysis performs pesticide residue studies for several of the largest domestic and foreign chemical producers to support new product registrations.

In most cases, complex method development and validation trials to determine the most effective extraction and instrumentation techniques were provided by ChemAllysis as part of the project. Since most of these studies are for new product registrations, details on compound names and results can not be divulged.

American Cyanamid Company - Princeton, NJ

ChemAllysis is currently involved in several registration studies for this client, including long term soil dissipation studies. Approximately 10,000 analyses will be performed by the end of these studies.

Pennwalt Corporation - Bryan, TX

ChemAllysis developed and validated the soil analysis method which is currently being utilized to analyze freezer stability during this 18 month study.

Cadbury & Schweppes Inc., Trumbull, CT

ChemAllysis has performed pesticide residue screens and specific pesticide assays on thousands of fresh apples and apple products. The project is on-going.

Ciba-Geigy Corporation - Greensboro, NC

ChemAllysis is currently working on several registration studies for Ciba-Geigy utilizing a number of GC protocols. For each of these projects, ChemAllysis submits

chromatographic data weekly with full reports at the end of each sampling period.

The Great Atlantic & Pacific Tea Co.,
Inc., Montvale, NJ

ChemAlysis has performed multiresidue pesticide screens on thousands of fresh fruits and vegetables. Project is on-going.

Hoechst Roussell Agri-Vet Company -
Somerville, NJ

The Company has been providing Hoechst with analytical services for several years. Thousands of samples have been analyzed for a variety of compounds using numerous extraction and instrumentation methods.

Monsanto Agricultural Co., St. Louis, MO

ChemAlysis is doing pesticide residue assays on grain matrices and method validation work.

II. ENVIRONMENTAL MONITORING

Rapid Turnaround Priority Pollutant Laboratory
Analytical Services

Maryland Department of the Environment-
Baltimore, MD

ChemAlysis was awarded a major contract to analyze samples collected by the State for full priority pollutants. Wastewater effluent, lake, river, and stream water collected throughout Maryland are analyzed for volatile organics, acid/base/neutrals, pesticides, PCBs, and 13 metals. Results for all analyses were reported within 7 days of sample submittal.

Analysis of Environmental, Waste Water,
and Drinking Water Samples for Volatiles
and Trace Metals

Honeywell Corporation - Annapolis, Maryland

ChemAllysis analyzes groundwater, wastewater, soils, and sediment for volatile organics and the drinking water metals by EPA methods 601, 602, 624, and Atomic Absorption techniques. A turnaround of 24 hours for the volatiles and

seven days for the metals is frequently required.

Analysis of Water Pollutants

Commonwealth of Virginia, State Water Control Board - Richmond, VA

ChemAllysis performs priority pollutant analyses for surface water and effluent discharge samples collected continuously from sites located throughout the State of Virginia. Requirements include meeting strict deadlines and providing computerized reports for entry into a historical data base.

Waste Oil Analysis

US Army - Fort George G. Meade, MD

ChemAllysis has been awarded a contract to analyze waste oil at the Fort Meade Army Base in central Maryland. ChemAllysis has developed a schedule with army personnel to analyze the oil for PCBs and a variety of inorganic compounds.

Laboratory Analysis of Soils/Sediments and Water Quality

US Army Corps of Engineers - Baltimore District

ChemAllysis manages a five year contract to perform sampling, analysis, and evaluation/characterization of soils, sediments, and water collected at various dredging sites within the Baltimore district. Functions include scheduling sampling collection, inorganic and organic laboratory analyses, and presentation of findings for each task order delivered by the Corps.

Analysis of Soil and Groundwater Samples for Contamination

Dames and Moore - Bethesda, MD

ChemAllysis was selected as the laboratory on a soil and groundwater investigation prior to a large construction

effort in Northern Virginia. Samples were analyzed for the full list of EPA Priority Pollutants, Benzene/Toluene/Xylene (BTX), pH, sulfate, chloride, EP Toxicity extraction for metals, and flashpoint.

Analysis of Bulk Samples for Organics and Inorganics

IBM Corporation - Manassas, VA

ChemAllysis was awarded a contract to analyze bulk building materials for volatile organics, solvents, EP Toxicity metals, and other inorganic compounds. A turnaround time of 72 hours was maintained throughout the project.

Determination of Pollutants in Water and Extracted Bulk Solid Material

Consultech Engineering - Washington, D.C.

The purpose of this study was to investigate the presence of styrene and phthalates extracted from bulk solid particles. Detection and confirmation were accomplished using EPA methods 624 and 625. A comprehensive QA/QC program was developed at the client's request to ensure the legal defensibility of the results.

Emergency Response Analytical Services

Arnold and Porter - Washington, D.C.

A law firm representing a large consumables corporations has retained ChemAllysis for emergency response analytical services. The projects have included performing analyses to determine product safety, investigate product tampering, and measure EPA and FDA regulatory compliance concerning the importation of raw materials.

PCB Determination in Various Matrices

Westinghouse Engineering Services - Columbia, MD

ChemAllysis utilizes gas chromatography (GC/ECD) methods to analyze two proprietary materials for PCB content. Because of the nature of PCB removal, extremely rapid turnaround is provided for most projects.

Analysis of Industrial Hygiene Samples for Hydrocarbons

Apex Environmental - Bethesda, Maryland

Air samples from numerous industrial settings were analyzed for low-boiling hydrocarbons. ChemAnalysis utilized NIOSH methods to provide these industrial hygiene analyses.

KEY PERSONNEL

AR302199

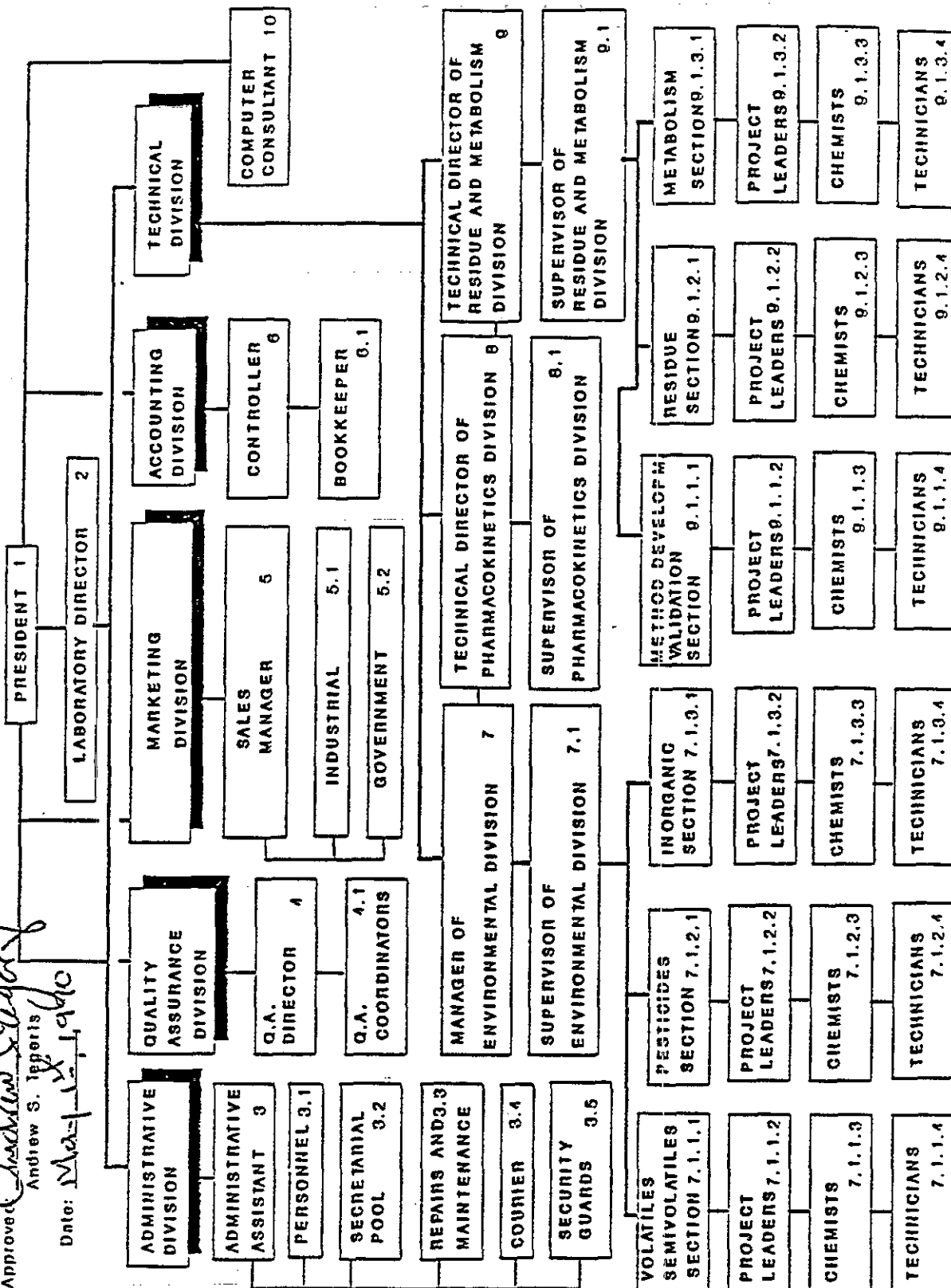
KEY PERSONNEL

Because a laboratory's most important asset is its personnel, Chemalysis has assembled an experienced, qualified staff with in-depth management, scientific and technical expertise. Currently numbering twenty eight employees, the Company has established an organizational structure designed for responsiveness and accuracy. Senior personnel coordinate all studies and provide management and technical guidance. A project leader is assigned to each job and is responsible for ensuring its timely, accurate completion. The Q.A./Q.C. Unit reviews all projects for EPA/CLP/GLP/FDA compliance, and reports its findings directly to the President.

Personnel profiles for top management, project leaders and the Q.A./Q.C. Director are provided on remaining following pages.

CHEMALYSIS ORGANIZATIONAL CHART

Approved: *Andrew S. Ippertis*
 Andrew S. Ippertis
 Date: *May 1, 1960*



CHEMALYSIS 1990 ORGANIZATIONAL CHART

President

1 Dr. Andrew S. Tegeris

Laboratory Director

2 Dr. Kathryn S. Kalasinsky

Administrative Division

3 Sherry A. Cipperly, Administrative Assistant

3.1 Personnel

3.2 Laura Becraft, Secretary/Receptionist

3.3 Chasie Keith, Maintenance & Repairs

3.4 Charles Gregonis, Raymond Shipley, Ronald Blair, Courier

3.5 Charles Fox, Security Guard

O.A. Division

4 Charles Tribett, B.S., M.S., Director

4.1 Marie Silverman, Coordinator

Marketing Division

5 Dr. Thomas EK, Sales Manager

Accounting Division

6 James Sokolis, Controller

6.1 Thomas Davey, Bookkeeper

Technical Division - Environmental

7 John F. Pfaff, M.S., Manager of Environmental Division

7.1 Supervisor Environmental Division

7.1.1.1 - Volatiles/Semivolatiles Section

7.1.1.2 - Project Leaders

7.1.1.3 - Chemists

7.1.1.4 - Technicians

7.1.2.1 - Pesticides Section

7.1.2.1 - Project Leaders

7.1.2.3 - Chemists

7.1.2.4 - Technicians

7.1.3.1 - Inorganics Section

7.1.3.2 - Project Leaders

7.1.3.3 - Chemists

7.1.3.4 - Technicians

Technical Division - Pharmacokinetics

8 Technical Director of Pharmacokinetics Division

8.1 Supervisor of Pharmacokinetics Division

Technical Division - Residue & Metabolism

9 Dr. Anthony Grigor, Technical Director, Residue & Metabolism Division

9.1 Supervisor of Residue Division

9.2 Kathy Morin, Asst. Supervisor of Residue Division

Computer Consultant

10 Thanasis Himonas, Computer Consultant

Chemists

John Pfaff, M.S.
Kathy Morin, B.S.
Michael Albertson, B.S.
Gregory Allen, B.A.
David Cabiles, B.S.
Mary Hagerman, B.A.
Geoff Maloney, B.S..
Catherine Reiter, B.A.
Andrea Ries, B.S.
Donna Davis, B.S.

Technicians

Peter Akinwumi, B.A.
Violeta Burgos, B.S.
Felecia Fort, B.A.
Emily Landis, M.S.
Leela Mathai, B.S.
Donald Shelly, B.S.
Matthew Ware, B.S.
Chris Hernandez, B.S.
Joyce Johnson
Ruth Ramseur

Weekend Technicians

Deore Dadaji, B.S.
Grant Kissel
Yan Lau, B.S.

ANDREW S. TEGERIS, M.D.
President

Education

Pathology Residency, Pathologic Anatomy, National Institutes of Health, Bethesda, MD, 1960 to 1961
Pathology Residency, Clinical Pathology, National Institutes of Health, Bethesda, MD, 1956 to 1957
Medical Internship, Duke University Hospital, Durham, NC, 1955 to 1956
M.D. Medical College of Virginia, 1955
B.S. Chemistry, College of William and Mary, 1951

Board Certifications

American Board of Toxicology, 1981
American Board of Pathology, 1961

Experience

1988 to Present: President, Chemalysis Incorporated, Laurel, MD.

Responsible for the overall administration and performance of analytical laboratory corporation. Chemalysis is a certified, GLP analytical laboratory supporting public and private sector clients in compliance with FIFRA, CERCLA, TOSCA, RCRA and NPDES regulations. Pursues new business through extensive network of contacts and professional colleagues. Oversees corporate financial and legal affairs. Provides direction for future growth, investigates new business areas.

1969 to 1987: President, Tegeris Laboratories, Inc., Laurel, Temple Hills, MD.

Conceived Tegeris Laboratories, Inc. as a large-scale animal toxicology research company. Designed, staffed, equipped, and directed two facilities performing long term animal toxicity studies for industrial and government clients. Actively participated in all projects including gross necropsies and histopathological evaluation of tissues. Well versed in the technical activities of the laboratory and managerial methods of operation.

1964 to 1975: Associate Clinical Professor of Pathology, Georgetown University School of Medicine and Dentistry, Washington, D.C.

1971 to 1975: Laboratory Director, Calvert County Hospital, Prince Frederick, MD.

1967 to 1969: Consultant Pathologist, Veterans Administration Hospital, Washington, D.C.

1961 to 1967: Clinical Pathologist, Division of Pharmacology,
U.S. Food and Drug Administration, Washington, D.C.

Professional Affiliations

National Agricultural Chemical Association
National Association of Life Science Industries
Society of Toxicology
American Society of Clinical Pathologists
College of American Pathologists
Medical Society of the State of Maryland
Society of Pharmacological and Environmental Pathologists
American Association for Laboratory Animal Science
Phi Beta Kappa
Alpha Omega Alpha

Publications

1. Tegeris, A.S.: Mechanism of hemolysis: In vitro effect of and primaquine and menadione on TPNH methemoglobin reductase; comparative studies on blood from man, dog, and swine, *Toxicol. and Appl. Pharmacol.* 6:361 (1965).
2. Tegeris, A.S., Curtis, H.M., Earl, F.L., and Smalley, H.E.: Metyoxychlor Toxicity: Comparative Studies in the Beagle Dog and Miniature Swine, *Toxicol. and Appl. Pharmacol.* 7:500 (1965).
3. Tegeris, A.S., Curtis, H.M., Earl, F.L., and Smalley, H.E.: Ornithine Carbamyl Transferase as a Liver Function Test: Comparative Toxicity in the Beagle Dog, Miniature Swine, and Man, *Toxicol. and Appl. Pharmacol.* 8:358 (1966).
4. Libre, P., Whang, J., Tegeris, A.S., and Holly, P.: Asymptomatic Megaloblastic Erythropoiesis with Chromosomal Abnormalities, *Clin. Res.* 14:310 (1966).
5. Tegeris, A.S., Cotrell, J.A., and Cruz, E.A.: The Effect of Some Commonly Prescribed Drugs on Routine Laboratory Procedures, *Abstr. Soc. Tox. No. 14*, p.11 (1969).
6. Tegeris, A.S., and Panteleakis, P.N.: The use of Females in Clinical (Human) Bioavailability Studies: the Effect of Menstruation, *Abstr. Soc. Tox., XII Ann. Mtg., Abstr. No. 150*, p.117 (1973).
7. Earl, F.L., Tegeris, A.S., Whitmore, G.E., Morrison, E., and Fitzhugh, O.G.: The use of Swine in Drug Toxicity Studies, *Ann. N.Y. Acad. Sci.* 3:671-688 (1964).
8. Tegeris, A.S.: Effect of Sodium Nitrite, Primaquine, and Menadione on TPNH Methemoglobin Reductase in Vitro, *Toxicol. and Appl. Pharmacol.*, 8:6-12 (1966).
9. Tegeris, A.S., Earl, F.L., and Curtis, J.M.: Normal Hematological and Biochemical Parameters of the Adult

Miniature Swine. In Swine in Biomedical Research: Proceedings of an International Symposium sponsored by Battelle Memorial Institute and the U.S. Atomic Energy Commission, July, 1965. Frayn Publishing Co., Seattle, WA, pp. 575-596 (1966).

10. Tegeris, A.S., Van der Weide, G., and Curtis, J.M.: Progressive Ultra Structural Changes in the Mucosa of the Small Intestine of the Beagle Dog Fed Methoxychlor, Expt, and Mol. Path. 18:243-258 (1968).
11. Tegeris, A.S., Smalley, H.E., and Curtis, J.M.: Ornithine Carbamyl Transferase as a Liver Function Test: Comparative Studies in the Swine and Man, Toxicol. and Appl. Pharmacol. 14:54-68 (1969).
12. Stanton, M.F., Layard, M., Tegeris, A.S., Miller, E., May, M., and Kent, E.: Carcinogenicity of Fibrous Glass: Pleural Response in the Rat in Relation to Fiber Demension, JNCI 58:587-603 (1977).
13. Stanton, M.F., Layard, M., Tegeris, A.S., Miller, E., May, M., Morgan, E., and Smith, A.: Relation of Particle Size to Carcinogenicity of Amphibole Asbestos and other fibrous materials, JNCI 67:965-976, (1981).
14. Tegeris, A.S.: Toxicology Laboratory Design and Management for the 80's and Beyond, S. Karger Publishing Co., New York, (1984).

KATHRYN S. KALASINSKY
Laboratory Director

Education

Ph.D. Analytical Chemistry, University of South Carolina, 1988
M.S. Physical Chemistry, University of South Carolina, 1976
B.S. Chemistry, University of South Carolina, 1974

Honoraries

Sigma Xi Honorary Research Society of North America, Outstanding Young Women of America, Omicron Delta Kappa Honorary Leadership Society, Who's Who in the South and Southwest, Personalities of the South, Most Outstanding Non-Teaching Professional Woman at MSU Awarded by the President's Commission on the Status of Women.

Professional Affiliations

American Chemical Society
Coblentz Society
Mississippi Academy of Science
American Association for the Advancement of Science
American Society for Testing and Materials (FT-IR Task Force)
Association of Official Analytical Chemists

Experience

September, 1989 to Present: Laboratory Director, ChemAnalysis Incorporated, Laurel, MD.

January, 1989 to September, 1989: Director, Spectroscopy, Mississippi State Chemical Laboratory.

1987 to 1988: Associate Director, Research Spectroscopy, Mississippi State Chemical Laboratory.

1983 to 1987: Assistant Director, Research Spectroscopy, Mississippi State Chemical Laboratory.

1981 to 1983: Chief Spectroscopist, Mississippi State Chemical Laboratory.

1977 to 1981: Research Spectroscopist, Mississippi State Chemical Laboratory.

1976 1977: Research Technologist II, Department of Food Science, Clemson University.

1974 to 1976: Research Assistant, Department of Chemistry, University of South Carolina.

Other Professional Activities

1. Chairman, 1983 Imported Fire Ant Conference.
2. Program Committee member for the Federation of Analytical Chemistry and Spectroscopy Societies (FACSS) meetings in Philadelphia, PA in 1981 and 1982.
3. Editor of the MSU Chemical Sciences Newsletter, 1979.
4. Panel member for the National Science Foundation (NSF) sponsored Women in Science Workshop in 1980 and 1981.
5. Consulting contracts with Pennzoil Oil Co., Battelle Laboratories, and Nichols Research Corp.
6. Coblentz Society Nominating Committee member for 1982.
Coblentz Society Board Member 1985-1989 (one of eight nationally elected positions).
Coblentz Society Newsletter editor 1986-.
Coblentz Society Spectral Evaluations Committee Co-Chairman, 1987-.
Coblentz Society President 1989-1991.
7. Chaired technical sessions for FACSS national meetings, 1981 and 1982, Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, 1989, Ohio State Symposium on Molecular Spectroscopy, Plenary Session, 1989.
8. Invited speaker:
Society for Applied Spectroscopy meeting, May, 1982 and February, 1983.
American Society for Testing and Materials meeting, November, 1982.
American Chemical Society meeting, November, 1983, September, 1985, May, 1986, June, 1986.
9. American Society for Testing and Materials (ASTM) FT-IR task force member responsible for writing standards for infrared spectroscopy.
10. Society for Applied Spectroscopy Tour Speaker, 1984.
11. Reviewer for Analytical Chemistry, Journal of Chromatography (Symposium Volumes), Applied Spectroscopy, Journal of the Association of Official Analytical Chemists, Spectroscopy.

Contract Work Solicited/Performed as Principal Investigator

1. "Investigation of Pesticide Degradation Products by GC-IR and other Analytical Techniques", Mississippi Department of Agriculture - \$15,395.
2. "Spectroscopic Analytical Determinations of Industrial and Agricultural Samples", Mississippi Industrial and Agricultural Services Division - \$17,368.
3. "Cellulosic and Lignin Surface Characteristics of Pulps and Papers", Mississippi Forest Products Laboratory- \$1,307.
4. "Degradation Product Analysis of Dyes used as Pesticides", Mississippi Agriculture and Forestry Experiment Station - \$1,203.
5. "Analysis of Vinyl Surface Coatings", U.S. General Tire - \$2,804.
6. "Analysis of Natural Products used for Pesticides", U.S. Department of Agriculture - Boll Weevil Research Lab - \$939.
7. "Quantitative Determination of Petro-Chemical Carbon Atom Character by Raman Spectroscopy", Pennzoil Research Corp. - \$3,869.
8. "LC/FT-IR Accessory Development for Aqueous Reverse and Normal Phase HPLC", Nicolet Instrument Corp. - \$59,545.
9. "Quantitative Analysis of Organo-Silane Coating on Fillers", Mallinckrodt Inc. - \$2,975.
10. "Herbicide Identification and Spectroscopic Data Base Generation", Ciba-Geigy Corp. - \$93,157.
11. "Identification of Paper Mill Process Contaminates and Paper Process Stream Component Data Base Generation- Weyerhaeuser Co. - \$10,208.
12. "Analyses of Produce Samples for Pesticide Residues", The Great Atlantic and Pacific Tea Co., Inc. - \$38,190.
13. "Analyses of Apple Products for Alar, Captan and EBDC Residues", Cadbury Schweppes Beverages - \$157,620.
14. "Analytical Testing for a Field Residue Study, OMITE 30W on Raspberries", Uniroyal Chemical Co., Inc. - \$27,585.
15. "Analysis of Profenofos on Cotton and Cotton Fractions", Ciba-Geigy Corp. - \$32,175.

16. "Analysis of Prometryn Residues in Cotton Seed and Cotton Seed Fractions", Ciba-Geigy Corp. - \$27,370
17. "GC/MS Method Development for the Analysis of Metalaxyl and it's Acid Metabolites in Ground Water", Ciba-Geigy Corp. - \$15,000.
18. "Determination of Diazinon Residues in Sweet Corn and Rotational Crop Samples", Ciba-Geigy Corp. - \$40,940

Research and Collaborative Projects

When Dr. Kathryn S. Kalasinsky joined MSCL in 1977, she directed the vibrational spectroscopy laboratory. In 1979 she also took on the direction of the nuclear magnetic resonance laboratory. Upon her arrival, the vibrational spectroscopy laboratory functioned only as a part of the Research Division of MSCL. Under her direction the vibrational spectroscopy laboratory made major contributions to the analytical services and analytical method development divisions as well.

Dr. Kalasinsky's main efforts in the Research Division were to develop GC/FT-IR for the analysis of pesticide degradation products. This work involved building a data base particular to the compounds of interest. Dr. Kalasinsky's other work with pesticides involved analysis of pesticides adsorbed onto soils and clays and pesticides encapsulated into polymers using Diffuse Reflectance Infrared Fourier Transforms (DRIFT) Spectroscopy.

Dr. Kalasinsky also collaborated with other departments (Chemical Engineering and Forest Products Laboratory) at MSU in developing a quantitative method for determining cellulose and lignin on the surface of paper and wood fibers by using reflectance techniques in an FT-IR experiment. This analysis is in demand by the paper and pulp industry and was the topic of an invited paper at the 1981 National Meeting of the American Institute of Chemical Engineers.

Dr. Kalasinsky has also collaborated with a major oil industry in their efforts to find a better method of analysis for determining the quantitative carbon atom character in kerosene and other oil products. The method that Dr. Kalasinsky devised employs Raman spectroscopy interfaced to the computer system of the FT-IR spectrometer. She is seeking ASTM approval of this analysis so that all oil industries can use this method on a regular basis.

Dr. Kalasinsky has collaborated with the University of Mississippi Medical Center in a project concerning the biomedical analysis of malaria infected blood using Fourier transform nuclear magnetic resonance techniques. It is hoped

that the results from this work will give a better understanding and thus a better cure for the devastating effects of malaria on the blood.

Dr. Kalasinsky also collaborated on a project with the Biochemistry Department on the MSU campus and the Mississippi Agriculture and Forestry Experiment Station (MAFES) concerning the analysis of the degradation products of dyes used as pesticides. This project led to the development of an LC/FT-IR accessory which several instrument manufacturers are interested in marketing.

Dr. Kalasinsky recently developed a better technique for GC/FT-IR analysis and is currently seeking a method of analysis of water pollutants by vibrational spectroscopy.

While at MSCL, Dr. Kalasinsky received two distinctive international honors. First, she was chosen by the National Science Foundation as one of fifty American scientists to receive a travel grant to present some of her work at the Sixth International Conference on Raman spectroscopy celebrated in Bangalore, India on the 50th anniversary of the discovery of the effect. Secondly, she was chosen by NATO as one of ten American scientists to attend an Advanced Study Institute held in Florence, Italy where scientists from all NATO nations joined in an exchange of current technologies and advances in the field of Fourier transform infrared spectroscopy.

Since joining Chemalysis, Inc., Dr. Kalasinsky has directed all aspects of the laboratory functions. This includes 25 professionals and 10 support staff. The scientific work has primarily focused on pesticide residues and environmental pollutants and her proposal success rate has been near 100%. She continues to be solicited for book review articles and method development in the combined fields of spectroscopy, pesticides and pollutants.

Publications

1. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Spectra and Structure of Organophosphorus Compounds XIII. Microwave, Raman and Infrared Spectra of CH_3POF_2 ", J. Mol. Struct., 34, 9 (1976).
2. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Vibrational Spectra and Molecular Symmetry of Tetrasilylhydrazine and Tetrasilylhydrazine - d_{12} ", J. Mol. Struct., 35, 201 (1976).
3. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Vibrational Spectra and Potential Function for the Low-Frequency Bending Mode of Silyl Isothiocyanate and Silyl Isothiocyanate - d_3 ", J. Phys. Chem., 82, 438 (1978).

4. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Potential Function for the Low-Frequency Bending Mode of Silylisocyanate", J. Chem. Phys., 69, 918 (1978).
5. K.S. Kalasinsky, J.R. Durig, and V.F. Kalasinsky, "Potential Function for the Low-Frequency Bending Mode of Silylisocyanate", Proceedings of the Sixth International Conference on Raman Spectroscopy, Bangalore, India, September 4-9, 1978, Volume 2, E. Schmid, R.S. Krishnan, W. Kiefer, and H.W. Schrotter, eds., Heyden, Philadelphia, 1978, p. 34.
6. A.R. Garber, P.D. Ellis, K. Seidman, and K. Schade (Kalasinsky), "Semiempirical Theory of Magnetic Resonance Parameters. I. Theory of Chemical Shielding Constants, Using Gauge Invariant Atomic Orbitals, Parameterization, and Carbon - 13 Chemical Shifts", J. Mag. Res., 34, 1 (1979).
7. G.R. Lightsey, P.H. Short, K.S. Kalasinsky, and L. Mann, "Effect of Surface Chemistry of Cellulosic Fillers on the Properties of Polypropylene Composites", J. Miss. Acad. Sci., 24, 76 (1980).
8. K.S. Kalasinsky, Technical Report, "(DOD classified topic)", Scientific Services Agreement, Delivery Order #1576, Army Research Office, August 1980.
9. K.S. Kalasinsky, "GC-IR Applications in Pesticide Chemistry", Proc. Soc. Photoopt. Inst. Eng., 289, 156 (1981).
10. K.S. Kalasinsky, Technical Report, "(DOD classified topic)", Scientific Services Agreement, Delivery Order #0024, Army Research Office, August 1982.
11. K.S. Kalasinsky, "GC/FT-IR Applications in Pesticide Chemistry", J. Chrom. Sci., 21, 246 (1983).
12. V.F. Kalasinsky, J.A.S. Smith, and K.S. Kalasinsky, "Rapid and Convenient Sampling Accessory for Diffuse Reflectance Spectroscopy", Appl. Spectrosc., 39, 552 (1985).
13. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Microbore High-Performance Liquid Chromatography/Fourier Transform Infrared Interface for Normal- or Reverse-Phase Liquid Chromatography", Anal. Chem., 57, 1969 (1985).
14. K.S. Kalasinsky, "Applications of Vibrational Spectroscopy to the Study of Pesticides", in Chemical, Biological, and Industrial Applications of Infrared Spectroscopy, J.R. Durig, e.d., John Wiley & Sons Limited, Chichester, England, 1985, Ch. 12, pp. 277-298.

15. V.F. Kalasinsky, S. Pechsiri, and K.S. Kalasinsky, "Analysis of Terpenes by Capillary Gas Chromatography/Fourier Transform Infrared Spectroscopy", J. Chromatogr. Sci., 24, 543 (1986).
16. V.F. Kalasinsky, K.G. Whitehead, R.C. Kenton, J.A.S. Smith, and K.S. Kalasinsky, "HPLC/FT-IR Interface for Normal- and Reverse-Phase Analytical Columns", J. Chromatogr. Sci., 25, 273 (1987), Feature Article.
17. V.F. Kalasinsky, K.G. Whitehead, R.C. Kenton, and K.S. Kalasinsky, "Double Lightpipe System for Analytical GC/FT-IR and Spectral Libraries", Appl. Spectrosc., 42, 75 (1988).
18. K.S. Kalasinsky, "Pesticide Determination by GC/FT-IR", in Analytical Methods for Pesticides and Plant Growth Regulators, Vol. XVI, J. Sherma, ed., Academic Press, Inc., San Diego, California, 1989, Ch. 4, pp. 101-117.
19. K.S. Kalasinsky, J.P. Minyard, Jr., V.F. Kalasinsky, and J.R. Durig, "Quantitative Analysis of Kerosenes by Raman Spectroscopy", Energy & Fuels, 3 (3), 304 (1989).
20. K.S. Kalasinsky, P.R. Griffiths, D.F. Gurka, S.R. Lowry, and M. Boruta, "Coblentz Specifications for Infrared Spectra of Materials in the Vapor Phase above Ambient Temperatures", Appl. Spectrosc. 44, 211 (1990).
21. K.S. Kalasinsky and J.R. Durig, "Industrial Applications in Vibrational Spectroscopy", Trends Anal. Chem., 9 (3), 83 (1990).
22. K.S. Kalasinsky, G.R. Lightsey, P.H. Short, and J.R. Durig, "Quantitative Analysis of Cellulosic Fillers Using Infrared Reflectance Spectroscopy", Appl. Spectrosc., 44, 404 (1990).
23. K.S. Kalasinsky, E.G. Alley, D.K. Cassell, and J.R. Durig, "Vibrational Frequency Patterns for Hydrogen Substituted Degradation Products of the Pesticide Mirex", Vibrat. Spectrosc., submitted for publication.
24. K.S. Kalasinsky and V.F. Kalasinsky, "Design and Applications of HPLC/FT-IR", in HPLC in the Pharmaceutical Industry, G. Fong, ed., Marcel Dekker, New York, 1990, in press.
25. J.R. Durig, J.F. Sullivan, D.T. Durig, and K.S. Kalasinsky, "Low Frequency Vibrational Spectra of Molecular Crystals XIII. Bromocyclopentane-d and Bromocyclopentane-d", Mol. Cryst. Liq. Cryst., manuscript in preparation.

Presentations

1. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Vibrational Spectra and Molecular Symmetry of Tetrasilylhydrazine and Tetrasilylhydrazine - d_{12} ", 31st Symposium on the Molecular Structure and Spectroscopy, Ohio State University, Columbus, OH, June 14-18, 1976.
2. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Potential Functions for the Low-Frequency Bending Modes of Silylisocyanate and Silylithiocyanate", 32nd Symposium on Molecular Structure and Spectroscopy, Ohio State University, Columbus, OH, June 13-17, 1977, presented by V.F. Kalasinsky.
3. J.R. Durig, K.S. Kalasinsky, and V.F. Kalasinsky, "Potential Function for the Low-Frequency Bending Mode of Silylisocyanate", 6th International Conference on Raman Spectroscopy, Bangalore, India, September 4-9, 1978.
4. K.S. Kalasinsky and E.G. Alley, "Capabilities of Fourier Transform - Infrared (FT-IR) Spectroscopy as Applied to Mirex Chemistry", 43rd Mississippi Academy of Sciences Meeting, Jackson Hilton Hotel, Jackson, MS, March 7-9, 1979.
5. G.R. Lightsey, P.H. Short, K.S. Kalasinsky, and L. Mann, "Effect of Cellulosic Filler Surface Chemistry on the Properties of Polypropylene Composites", 43rd Mississippi Academy of Sciences Meeting, Jackson Hilton Hotel, Jackson, MS, March 7-9, 1979, presented by G.R. Lightsey.
6. K.S. Kalasinsky, "GC-IR Applications in Pesticide Chemistry", 1979 FT-IR User's Conference, Nicolet Instrument Corp., Madison, WI, October 1-4, 1979.
7. K.S. Kalasinsky and G.R. Lightsey, "Quantitative Analysis of Cellulosic Filler Surface Chemistry Using Reflectance Spectroscopy and Computer Spectral Addition", 1980 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 10-14, 1980.
8. K.S. Kalasinsky and E.G. Alley, "Applications of FT-IR (Fourier Transform - Infrared) Spectroscopy to Insect Research", Imported Fire Ant Conference, Doyle Conner Building, Gainesville, FL, March 25-27, 1980.
9. E.G. Alley, B.R. Layton, J. Eubanks, J.L. Smathers, R.R. Ingram, and K.S. Kalasinsky, "Synthesis and Identification of Dihydrogen Derivatives of Mirex", 2nd Chemical Congress of the North American Continent, Las Vegas, Nevada, August, 1980, presented by E.G. Alley.

10. K.S. Kalasinsky and G.R. Lightsey, "Quantitative Analysis of Cellulosic Filler Surface Chemistry Using Infrared Reflectance Spectroscopy and Computer Spectral Addition", 1980 Nicolet FT-IR User's Conference, Nicolet Instrument Corp., Madison, WI, September 15-18, 1980.
11. K.S. Kalasinsky, "Quantitative Analysis of Kerosenes by Raman Spectroscopy", 1981 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 9-13, 1981.
12. K.S. Kalasinsky, "GC-IR Applications in Pesticide Chemistry", 1981 International Conference on Fourier Transform Infrared Spectroscopy, University of South Carolina, Columbia, South Carolina, June 8-12, 1981.
13. K.S. Kalasinsky, "GC-IR Applications in Pesticide Chemistry", 8th Annual Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Philadelphia, PA, September 20-25, 1981.
14. K.S. Kalasinsky, "Quantitative Analysis of Kerosenes by Raman Spectroscopy", 8th Annual Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Philadelphia, PA, September 20-25, 1981.
15. K.S. Kalasinsky, "You Want an Infrared Spectrum of What?!", 1981 Nicolet FT-IR User's Conference, Madison, WI, October 5-8, 1981.
16. P.H. Short, G.R. Lightsey, and K.S. Kalasinsky, "Effect of Refining Time and Temperature Variables on the Surface Chemistry of Fiberboard Furnish", 1981 American Institute of Chemical Engineers Annual Meeting, New Orleans, LA, November 8-12, 1981, presented by P.H. Short.
17. K.S. Kalasinsky, "GC/FT-IR in Pesticide Chemistry", Mid-South Chromatography Symposium", Memphis, TN, November 12-13, 1981. (Invited)
18. K.S. Kalasinsky and J.T. McDonald, "Developments in Chromatographic Infrared Techniques for the Analysis of Pesticide Degradation Products", 1982 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 8-13, 1982.
19. K.S. Kalasinsky, R.R. Ingram, and J.L. Smathers, "Field Trials on EL-468 and Amdro", 1982 Imported Fire Ant Conference, Austin, TX, March 25-26, 1982, presented by J.L. Smathers.
20. K.S. Kalasinsky, "GC-IR Applications in Pesticide Chemistry", 1982 Society for Applied Spectroscopy Multi-Discipline Symposium, Chicago, IL, May 3-6, 1982. (Invited)

21. K.S. Kalasinsky, J.T. McDonald, and V.F. Kalasinsky, "Developments in Chromatographic Infrared Techniques for the Analysis of Pesticide Degradation Products", 9th Annual Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Philadelphia, PA, September 19-24, 1982.
22. K.S. Kalasinsky, "Developments in Chromatographic Infrared Techniques", 1982 Nicolet FT-IR User's Conference, Madison, WI, October 4-7, 1982.
23. K.S. Kalasinsky, "Developments and Applications of HPLC/IR", 21st Annual Meeting on the Practice of Chromatography, New Orleans, LA, October 10-13, 1982 (Invited)
24. K.S. Kalasinsky, "Applications of FT-IR in Pesticide and Analytical Chemistry", Society of Applied Spectroscopy, Chicago Regional Meeting, Chicago, IL, February 9, 1983. (Invited)
25. K.S. Kalasinsky, J.T. McDonald, and V.F. Kalasinsky, "Aqueous Reverse Phase LC/FT-IR Developments and Applications for the Analysis of Pesticide Degradation Products", 1983 Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 7-11, 1983.
26. K.S. Kalasinsky, "You Want an FT-IR Spectrum of What?!", 1983 DATA Group Symposium, Jackson, MS, April 26, 1983. (Invited)
27. K.S. Kalasinsky and V.F. Kalasinsky, "Pesticide Degradation Analysis by Chromatographic Infrared Techniques", poster presentation, 13th Annual Symposium on the Analytical Chemistry of Pollutants, Jekyll Island, GA, May 16-18, 1983.
28. K.S. Kalasinsky, J.T. McDonald, and V.F. Kalasinsky, "Aqueous Reverse Phase LC/FT-IR Developments and Applications", 25th Rocky Mountain Conference, Denver, CO, August 14-17, 1983.
29. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Aqueous Reverse Phase LC/FT-IR Developments and Applications", 1983 FT-IR User's Conference, Madison, WI, October 3-6, 1983.
30. K.S. Kalasinsky, "Chromatographic Infrared Applications in Pesticide Chemistry", 1983 Southeast Regional Meeting of the American Chemical Society, Charlotte, NC, November 9-11, 1983. (Invited)
31. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Aqueous Reverse Phase LC/FT-IR Developments and Applications", Eastern Analytical Symposium, Inc., New York City, NY, November 16-18, 1983. (Invited)

32. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Aqueous Reverse Phase LC/FT-IR", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 5-9, 1984.
33. K.S. Kalasinsky, "Applications of FT-IR in Pesticide and Analytical Chemistry", Society of Applied Spectroscopy National Tour Speaker. Presented in Baton Rouge, LA, April 12, 1984; Albuquerque, NM, April 18, 1984; Houston, TX, April 19, 1984.
34. K.S. Kalasinsky, "You Want an Infrared Spectrum of What?!", Society of Applied Spectroscopy National Tour Speaker. Presented in Denver, CO, April 16, 1984; and in Tucson, AZ, April 17, 1984.
35. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Developments in Aqueous Reverse Phase LC/FT-IR", Eastern Analytical Symposium, New York, NY, November 13-16, 1984, presented by J.A.S. Smith.
36. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "Applications of Reverse Phase LC/FT-IR", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, New Orleans, LA, February 25-March 1, 1985, presented by J.A.S. Smith.
37. J.A.S. Smith, K.S. Kalasinsky, and V.F. Kalasinsky, "Aqueous Reverse Phase HPLC/FT-IR Spectroscopy", 7th Mid-Winter Symposium, Mississippi Section - American Chemical Society, Columbus, MS, March 21, 1985, presented by J.A.S. Smith.
38. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "A HPLC/FT-IR Interface for Use with Normal or Reverse Phase Chromatography", National American Chemical Society Meeting, Chicago, IL, September 8-13, 1985, presented by V.F. Kalasinsky.
39. K.S. Kalasinsky, "Chromatographic Infrared Developments and Applications to Pesticide Chemistry", American Chemical Society Cincinnati Section Symposium, Cincinnati, OH, September 19, 1985. (Invited)
40. K.S. Kalasinsky, J.A.S. Smith, and V.F. Kalasinsky, "An HPLC/FT-IR Interface for Use with Normal or Reverse Phase Chromatography", Southeast Regional American Chemical Society Meeting, Memphis, TN, October 9-11, 1985, presented by V.F. Kalasinsky.

41. V.F. Kalasinsky, J.A.S. Smith, K.S. Kalasinsky, and K.G. Whitehead, "Advances in Aqueous Reverse Phase LC/FT-IR", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 10-14, 1986, presented by K.G. Whitehead.
42. V.F. Kalasinsky, J.A.S. Smith, K.S. Kalasinsky, and K.G. Whitehead, "Advances in Aqueous Reverse Phase HPLC/FT-IR", 8th Mid-Winter Symposium, Mississippi Section - American Chemical Society, Hattiesburg, Mississippi, April 10-11, 1986, presented by K.G. Whitehead.
43. K.S. Kalasinsky and V.F. Kalasinsky, "Applications of GC/FT-IR and HPLC/FT-IR", Louisiana Section - American Chemical Society Meeting, New Orleans, Louisiana, May 7-8, 1986. (Invited)
44. K.S. Kalasinsky and V.F. Kalasinsky, "Reverse Phase HPLC/FT-IR Designs and Applications", 1986 ACS Analytical Summer Symposium, University of Utah, Salt Lake City, Utah, June 18-20, 1986. (Invited)
45. K.S. Kalasinsky, K.G. Whitehead, R.C. Kenton, and V.F. Kalasinsky, "Double Lightpipe System for Analytical GC/FT-IR and Spectral Libraries", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 9-11, 1987.
46. V.F. Kalasinsky, K.G. Whitehead, R.C. Kenton, and K.S. Kalasinsky, "Reverse Phase HPLC/FT-IR of Pharmaceuticals and Organic Acids", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlantic City, NJ, March 9-13, 1987, presented by V.F. Kalasinsky.
47. R.C. Kenton, V.F. Kalasinsky, K.G. Whitehead, and K.S. Kalasinsky, "Reverse Phase HPLC/FT-IR of Pharmaceuticals and Organic Acids", 9th Mid-Winter Symposium, Mississippi Section - American Chemical Society, Mississippi State, MS, March 26-27, 1987, presented by R.C. Kenton.
48. K.G. Whitehead, V.F. Kalasinsky, K.S. Kalasinsky, and R.C. Kenton, "Double Lightpipe System for Analytical GC/FT-IR and Spectral Libraries", 9th Mid-Winter Symposium, Mississippi Section - American Chemical Society, Mississippi State, MS, March 26-27, 1987, presented by K.G. Whitehead.
49. V.F. Kalasinsky, R.C. Kenton, K.G. Whitehead, and K.S. Kalasinsky, "HPLC/FT-IR Analysis by Diffuse Reflectance", 14th Annual Federation of Analytical Chemistry and Spectroscopy Societies Meeting, Detroit, MI, October 4-9, 1987, presented by V.F. Kalasinsky.

50. V.F. Kalasinsky, R.C. Kenton, K.G. Whitehead, and K.S. Kalasinsky, "Developments and Applications of HPLC/FT-IR", 39th Southeast Regional Meeting of the American Chemical Society, Orlando, FL, November 3-6, 1987, presented by V.F. Kalasinsky.
51. K.S. Kalasinsky, "Industrial and Agricultural Applications in the Vibrational Spectroscopy Laboratory", University of South Carolina, Department of Chemistry Seminar, Columbia, SC, June 21, 1988.
52. K.S. Kalasinsky, "Chromatographic Infrared Techniques and Applications in Analytical Chemistry", National Bureau of Standards, Analytical Division Seminar, Gaithersburg, MD, June 27, 1988.
53. K.S. Kalasinsky, "Industrial and Agricultural Applications of Vibrational Spectroscopy", University of South Carolina, Department of Chemistry Seminar, Columbia, SC, November 17, 1988.
54. V.F. Kalasinsky, T.H. Pai, R.C. Kenton, and K.S. Kalasinsky, "Gradient Elution Aqueous Reversed-Phase HPLC/FT-IR Using Immobilized Post-Column Dehydration Reactions", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Atlanta, GA, March 6-10, 1989, presented by T.H. Pai.
55. K.S. Kalasinsky, "Industrial Applications of Vibrational Spectroscopy", First Annual Proctor & Gamble Vibrational Spectroscopy Symposium, Cincinnati, Ohio, June 16, 1989 (Invited).
56. V.F. Kalasinsky, T.H. Pai, R.C. Kenton, and K.S. Kalasinsky, "Aqueous Reversed-Phase HPLC/FT-IR Using Diffuse Reflectance Detections", poster presentation, 7th International Conference on Fourier Transform Spectroscopy, Fairfax, VA, June 19-23, 1989.
57. V.F. Kalasinsky and K.S. Kalasinsky, "HPLC/FT-IR: Design and Applications", SmithKline French Research & Development Seminar, King of Prussia, PA, Nov. 8, 1989, presented by V.F. Kalasinsky (Invited).
58. T.H. Pai, V.F. Kalasinsky, and K.S. Kalasinsky, "Developments and Applications of Vibrational Reflection-Absorption Spectroscopy", Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, New York City, NY, March 5-9, 1990, presented by V.F. Kalasinsky.

ANTHONY F. GRIGOR, Ph.D.
Technical Director, Residue & Metabolism Division

Education

Ph.D. Chemistry, The Pennsylvania State University, 1966
B.S. Chemistry, King's College, 1961

Experience

1988 to Present: Technical Director, Residue & Metabolism Division, ChemAllysis Incorporated, Laurel, MD.

Responsible for technical management of complex pesticide residue and hazardous waste contamination studies. Supervises a large staff of chemists and chemical technicians in the performance of sample preparation and instrumental analyses. Researches, conducts, and monitors development of new analytical methodologies or modification of existing procedures to meet study requirements. Responsible for constant monitoring of technical quality of all projects and reviewing reports before release.

1985 to 1988: Chemistry Director, Tegeris Laboratories Inc., Temple Hills, MD.

Directed the chemistry division in the conduct of numerous environmental and pesticide residue studies. Prepared reports for preparation to officials at the EPA and FDA. Reviewed data, provided technical support and direction.

1976 to 1985: Chief, Division of Laboratories, Pennsylvania Department of Agriculture, Harrisburg, PA.

Directed the operations of the Bureau of Foods and Chemistry Laboratory in the testing of foods, water, feeds, and fertilizers to ensure compliance with regulations concerning purity, adulterations, or contamination. Prepared annual budgets, justified new equipment purchases, and monitored expenditures. Significantly improved quality assurance review of all protocols and data. Developed a laboratory manual of all testing methodologies, established a comprehensive safety program, provided technical information to the public and interacted with FDA, EPA, and USDA.

1971 to 1976: Assistant Chief Chemist, Pennsylvania Department of Agriculture, Harrisburg, PA.

Managed food testing laboratory services. Designed and established the State's meat testing program. Utilized wet chemistry techniques as well as gas chromatography, ultraviolet, and atomic absorption methodologies.

1967 to 1971: Senior Research Chemist, Allegheny Ballistics Laboratory, Cumberland, MD.

Studied decomposition kinetics and catalysis of propellant ingredients by time-flight mass spectrometry, infrared analysis, and thermal analysis. Postulated mechanisms for the ingredient decompositions and interactions and recommended optimum catalysts.

Professional Affiliations

American Chemical Society
Association of Official Analytical Chemists
Institute of Food Technologists
Association of Food and Drug Officials

Publications

1. The Measurement and Correlation of Some Physical Properties of CH₄ and CD₄: Ph.D. Thesis, 1966.
2. Grigor, A.F. and Steele, W.A.: Density of Balance for Low Temperatures and Elevated Pressures, Rev. Sci. Instr. 37, 51-54, 1966.
3. Grigor, A.F. and Steele, W.A.: Physical Properties of Fluid CH₄ and CD₄: Experimental, J. Chem. Phys. 48: 1032-1037, 1968.
4. Grigor, A.F. and Steele, W.A.: Physical Properties of Fluid CD₄ and CD₄: Theory J. Chem. Phys. 48: 1038-1046, 1968.
5. Grigor, A.F. and Steele, W.A.: Critical Properties of Argon: Phys. and Chem. Liquids: 1:1-11, 1968.
6. Grigor, A.F. and Musso, R.C.: Decomposition Studies of Propellant Ingredients and Ingredient Combinations, AIAA Paper, 68-495, ICRPG/AIAA 3rd Solid Propulsion Conference, Atlantic City, N.J., June 1968.
7. 1968-1971: Wrote numerous progress reports on behalf of Allegheny Ballistics Laboratory to the funding agency, USAF.
8. 1971-1976: Wrote Standard Operating Procedures for the Meat Testing Laboratory of the State of Pennsylvania. Wrote numerous progress reports, including annual reports.

9. 1976-1985: Updated Standard Operating Procedures. Wrote budget requests and annual reports on the various projects to the respective funding agencies.
10. Grigor, A.F.: High pressure liquid chromatographic determination of Safarol in foods; in preparation.
11. Grigor, A.F.: High pressure liquid chromatographic determination of chlorpropham in potatoes; in preparation.
12. Grigor, A.F.: High pressure liquid chromatography of sodium benzoate and potassium sorbate in meats; in preparation.
13. Grigor, A.F.: Determination of alcohol in wine and candy by high pressure liquid chromatography; in preparation.
14. Grigor, A.F. and Bemederfer, V.J.: Determination of the authenticity of peanut oil and peanut butter by gas chromatography; in preparation.

THOMAS P. EK
Marketing and Sales Manager

Education

Ph.D., Biomedical Science/Pharmacology, Northeastern University, Boston Massachusetts, 1990

B.S., Biology, Geneva College, Beaver Falls, Pennsylvania, 1985

Experience

January 1990 to present: Marketing and Sales Manager, ChemAllysis Inc., Laurel, Maryland

Has thorough understanding of ChemAllysis' Standard Operating Procedures and Strategic Plan; knows the nature of the company's business. Markets ChemAllysis' capabilities and expertise to industry and government.

1985 - 1987: Graduate Teaching Assistant, College of Pharmacy, Northeastern University, Boston, Massachusetts

1983 - 1985: Undergraduate Teaching Assistant (Philosophy): Geneva College, Beaver Falls, Pennsylvania

1979 - 1983 (Summers): Transportation Aide: (Central Service Department), Maine Medical Center, Portland, Maine

Publications:

1. Ek, T.P., and Deth, R.C. Elevated phospholipase C and Na⁺-H⁺ exchange activity in spontaneously hypertensive rats. Hypertension 12: 330-331, 1988.
2. Ek, T.P., Campbell, M.D., Jagadeesh, G., and Deth, R.C. Reduction of norepinephrine-induced tonic contraction and phosphoinositide turnover in arteries of spontaneously hypertensive rats: a possible role for Protein kinase C. Am. J. Hypertension 2: 40-45, 1989.
3. Ek, T.P., Danthuluri, N.R., and Deth, R.C. Effect of different buffers on norepinephrine-induced contractile response in spontaneously hypertensive rats: a possible role for the intracellular pH. (Submitted for publication)

Abstracts and Presentations:

1. Ek, T.P., Campbell, M.D., and Deth, R.C. Reduction in agonist-induced ^{32}P -phospholipid labelling and contractile response in SHR arteries. FASEB, Washington D.C. (1987)
2. Gupta, S., Ek, T.P., Cragoe, E.J. Jr., and Deth, R.C. Stimulation of Na^+/H^+ exchange by ANF in rabbit aorta. ASPET, Honolulu, Hawaii. (1987)
3. Ek, T.P., Gupta, S., and Deth, R.C. Differences in agonist-induced ^3H -myo-inositol labelling, contractile response, and Na^+/H^+ exchange in SHR vs WKY arteries. The Pharmacologist 30 (3):A164 (1988)
4. Ek, T.P., and Deth, R.C. Effect of different buffers on norepinephrine-induced contractile response in SHR. Meeting of New England Pharmacologists, Boston, Massachusetts. (1989)

CHARLES WILLIAM TRIBETT
Quality Assurance Director

Education

M.B.A. Business Administration, Wheeling College, 1984
B.S. Chemistry, West Liberty State College, 1960
B.S. Mathematics, West Liberty State College, 1960

Experience

April, 1989 to Present: Quality Assurance Director, ChemAllysis Incorporated, Laurel, MD.

Responsible for development, implementation, and management of ChemAllysis' comprehensive QA/QC program. Monitors operations in sample receiving and analysis to ensure adherence to all facets of the program. Implements corrective actions if inadequacies are revealed.

Performs critical reviews of all experimental data presented in the progress and final reports. Performs frequent quality assurance inspections to detect any deviations in the analytical procedures or methods dictated by protocols. Inspects facilities and checks records for equipment service, calibrations, and maintenance. Reports any deviations in methodologies, unacceptable conditions in the laboratory, and faulty equipment maintenance to the Company President.

1987 to 1989: Independent Contractor - Analytical Chemistry and Quality Assurance, WV and PA.

Provided support to such corporations as Allied-Signal, Rollins Environmental Services, and LCP Chemicals on environmental and industrial compliance issues. Directed large scale site investigation on industrial facility prior to closure. Coordinated sampling crews and managed on-site GC, AA, and HPLC laboratory.

1986 to 1987: Manager of Operations, Castamerica, Inc. Mount Lebanon, PA.

Directed the general operations of a industrial facility engaged in the manufacture of alkyd, furfaryl, urethane, phenolic, and urea-formaldehyde resins. Had overall responsibility for quality assurance, effluent and air discharge compliance, and storage and disposal of hazardous chemicals and wastes in addition to manufacturing.

1985 to 1986: Technical Director, Newell Specialty Chemicals, Inc., Newell, WV.

Responsible for the technical coordination of manufacturing activities with client companies. Directed quality control group, performed subcontracting and supervising research, explored process improvement, and managed regulatory affairs with EPA, FDA, and DOT.

1960 to 1984: Quality Assurance Manager, Manager of Distribution, Superintendent -Quality Control, Supervisor-Quality Control, Supervisor-Chemical Control, Analytical Chemist, Olin Corp./Allied Chemical Corp., Moundsville, WV.

In 24 years with Olin/Allied developed quality assurance manuals, designed specifications for purchased materials, intermediate and finished products, SQC and SPC programs. Was responsible for product distribution and accountability, employee training and safety, and relations with customers for matters concerning quality and service. Facility liason with EPA, DOT, FDA, and other federal and state regulatory agencies. Performed organic and inorganic syntheses and analysis by latest instrumental methodologies.

Professional Associations

American Society for Quality Control
American Society for Testing Materials
American Chemical Society

MARIE E. SILVERMAN, AALAS
Quality Assurance Coordinator

Education

Certification: AALAS, Certification as Assistant Laboratory Technician, 1974-1980

Franklin Regional High School, Murrysville, Pennsylvania, Business Major, 1963-1966

Education seminars for employees sponsored by Tegeris Laboratories, Inc. include: "Genetics and Nomenclature for the Non-Geneticist," speaker - Louis J. Serrano, D.V.M., Frederick Cancer Research Center, Frederick, Maryland; "Establishment of Gnotobiotic Inbred Mouse Colonies," speaker - Thomas W. Davis, D.V.M., Frederick Cancer Research Center, Frederick, Maryland; and "Present Status of Mutagenicity Testing," speaker - Marvin S. Legator, Ph.D., Division of Environmental Toxicology, The University of Texas Medical Branch, Galveston, Texas.

Experience

December 1986 - Present: Quality Assurance Coordinator, ChemAnalysis, Incorporated (formerly Tegeris Laboratories, Inc.), Laurel, MD

Inspects various aspects of laboratory experimentation, both historically (data inspection) and by direct observation, and documents for Quality Assurance Director the degree of adherence to the approved protocol, ChemAnalysis SOPs and the appropriate Federal Regulations or lack thereof.

Major responsibilities consist of, but are not limited to, the following: (a) Regularly inspects all experimental activities and documents all findings according to a schedule designed by the Quality Assurance Director. (b) Maintains the Archives Room with a filing and recall system, including the SOP file. (c) Reviews final reports for Quality Assurance.

Also maintains master file of ChemAnalysis SOPs and performs QA inspections accordingly. Maintains copies of current SOPs and of all active protocols. Performs periodic quality methods dictated by protocols. Performs critical reviews of all experimental data presented in progress and final reports to assure accuracy of description and completeness of data recorded therein. Inspects all laboratories for cleanliness and orderliness and checks records for equipment service, calibrations and maintenance. Reports any deviations in method of experimental, unacceptable conditions in laboratory and faulty equipment maintenance to Quality Assurance Director with a copy to the President or Laboratory Director. Maintains an inventory of raw data and final reports on all studies.

1977 - November 1986: Animal Technician/Junior Study Coordinator, Tegeris Laboratories, Inc., Laurel, MD

Responsibilities included: all phases of maintaining and caring of animals, observed for abnormalities and/or signs of toxicity, data taking, including body weight and feed consumption, familiarity and executing TL's Standard Operating Procedures and related sections of Sponsor protocols, assisted in collection of metabolic samples of blood, urine, and feces, monitored both temperature and humidity throughout the laboratory, maintained cleanliness of laboratory and animal quarters.

1974 - 1977: Assistant Laboratory Technician, University of Pittsburgh, School of Medicine, Surgery Department

Responsibilities included: received and vaccinated animals upon arrival, assisted in blood drawing, took fecal samples and did TB testing, knowledgeable of animal treatment, assisted in surgery, identified animals, recordkeeping, and maintained an inbred rat colony.

JOHN PFAFF
Manager, Environmental Services

Education

M.S. Environmental Science and Engineering, Virginia Polytechnic Institute, 1981
B.S. Biology, Virginia Polytechnic Institute, 1979
GC/MS Training Course.

Experience

May, 1988 to Present: Manager, Environmental Services,
ChemAllysis Incorporated, Laurel, MD.

Directs operations of the Company's Environmental Services Section providing instrumental analyses for priority pollutants, volatile organics, pesticides, PCBs, and metals. Responsible for scheduling, operating, and maintaining over \$400,000 of equipment including 3 GC/MSs, 7 GCs, 3 AAs, and other instrumentation. Extensive analytical experience in organic disciplines. Four years experience as GC/MS operator for priority pollutant analysis (EPA 624, 625), and five years experience with volatile organics analysis (EPA 601, 602).

Possesses substantial technical knowledge of civil and environmental engineering practices in water treatment, solid waste management, and pollutant abatement through graduate course work and field experience.

1983 to 1988: Section Leader, Analytical Services, Biospherics Incorporated, Beltsville, MD.

Managed field and laboratory environmental assessment programs involving water and biological sampling, fate and metabolism studies, distribution and run-off studies, leachability and adsorptivity measurements, and treatability studies. Operated GC/MS for priority pollutant and pesticide residue analyses. Managed GC laboratory analyzing volatile organics, PCB's, and pesticides. Integrated raw data into report formats and interacted frequently with clients. Monitored chemists and technicians to ensure that proper EPA and FDA methodologies were being followed.

Supervised ground water analytical program for an uncontrolled hazardous waste facility for 3 years. This involved the performance of 100-200 volatile organic analyses each month by EPA methods 601, 602, and 624 within a 20 day turnaround period. Monitored quality and validity of data and coordinated the flow of samples and results.

Responsible for several long term projects including:

- o Supervision of 200-300 volatile organic analyses monthly for several industrial clients involved in hazardous waste remedial actions
- o Management of thousands of organic priority pollutant, pesticide, and PCB analyses for a U.S. Air Force analytical services contract.
- o Performance of organic residue (growth regulator) in crop analyses by GC/MS for several major agricultural chemical manufacturers.

1982 to 1983: Environmental Chemist, JTC Environmental Consultants, Rockville, MD.

Analyzed pesticides, herbicides, explosives, malodorous compounds, and other residues by GC and HPLC. Also had responsibility for developing new analysis methodologies, total organic halide measurements, data interpretation, and training.

1979 to 1981: Graduate Student Research Assistant, Departments of Civil and Sanitary Engineering, Virginia Polytechnic Institute, Blacksburg, VA.

Professional Affiliations

American Chemical Society
National Water Well Association
Washington Chromatography Group

KATHY R. MORIN
Assistant Supervisor/Analytical Chemist

Education

B.S. Biochemistry, Albright College, 1987
"Developing Liquid Chromatography Separations", Waters Inc., 1989

Experience

September 1, 1989 to Present: Assistant Supervisor, Residue Section, ChemAllysis, Incorporated, Laurel, Maryland.

Besides her responsibilities as an analyst (see below), she helps with the supervision of analysts and technicians and with the management of their projects.

March, 1988 to August 31, 1989: Analytical Chemist/Project Leader, ChemAllysis Incorporated, Laurel, MD.

Directs complex projects for analysis of pesticide and drug residues in soil, crop, and animal tissues. Develops new methods to analyze pesticides and conducts validations. Performs quantitative analysis of residues at ppm and ppb level using gas chromatography and high pressure liquid chromatography. Schedules work flow, supervises sample extraction and preparation, and maintains equipment. Reduces raw data into report formats for submission to QA/QC. Follows all GLP protocols.

1987 to 1988: Chemist, Biospherics Incorporated, Beltsville, MD.

Prepared samples from a variety of plant, tissue, and environmental matrices for analysis. Supervised chemical technicians in wet laboratory techniques. Responsible for all wet laboratory work and report writing for \$900,000 pesticide registration project.

1985 to 1987: Laboratory Assistant, Chemistry Department, Albright College, Reading, PA.

Instructed students in laboratory methods, prepared and equipped labs for seminars, and maintained instrumentation.

Professional Affiliations

American Chemical Society

MARY HAGERMAN
Project Leader, Inorganics

Education

B.A. Chemistry, Earlham College, 1983

Experience

August, 1988 to Present: Project Leader, Inorganics, ChemAlysis Incorporated, Laurel, MD.

Responsible for establishing the inorganic laboratory and developing analytical and documentation procedures for all inorganic analyses. Supervises preparation of inorganic samples and performs trace metals analyses by atomic absorption (AA), graphite furnace, cold vapor, and hydride generation using the advanced Varian SpectrAA 400 system. Manages inorganic projects and prepares proposals for private and government contracts.

1987 to 1988: AA Group Leader, Environmental Science and Engineering, St. Louis, MO.

Supervised laboratory for trace metals and general inorganics. Established standard operating procedures and documentation for inorganics section of environmental laboratory. Analyzed environmental, industrial hygiene, and hazardous waste samples by graphite furnace, flame AA, and ion chromatography. Managed CLP analyses, RCRA testing, EP TOX/TCLP generation, cyanides, phenols, and other general chemistry parameters.

1984 to 1987: Assistant Metals Laboratory Director: JTC Environmental Consultants, Rockville, MD.

Supervised and coordinated chemists for work under EPA Inorganics Contract Laboratory Program (CLP). Responsible for contractual compliance, reports, budgets, and instrument trouble shooting. Managed analytical projects. Prepared EPA contract bids.

- o Trace Metals Analysis - Four years experience with ICAP and AA analysis for trace metals, graphite furnace, and atomic absorption for EPA CLP program and private clients.
- o High Concentration/Hazardous Waste Analysis - Analyzed for metals in hazardous waste for EPA and U.S. Navy using ICAP and hydride generation. Developed methods for handling and analyzing unknown chemical and organic wastes.

- o Explosives Desensitization Research - Studied RDX and TNT desensitization reaction rates using HPLC following U.S. Army Toxic and Hazardous Materials Agency regulations. Studied feasibilities of explosive removal from lagoons and sludges.
- o Pesticide and PCB Analysis - Analyzed environmental samples for pesticides and PCB's by gas chromatography.

MICHAEL ALBERTSON
Analytical Chemist

Education

B.S. Earth Sciences, Gannon University, 1986

Experience

May, 1988 to Present: Analytical Chemist, ChemAlysis Incorporated, Laurel, MD.

Exclusively dedicated to volatile organic analysis in matrices of ground water, waste water, soil, and air. Extensive experience analyzing thousands of environmental samples by EPA methods 601, 602, and 624. Expert knowledge of GC and GC/MS methodologies. Responsible for data reporting and equipment maintenance.

Extensive computer systems knowledge. Interfaces personal computers to chromatographic work stations to automate data retrieval from analytical instruments. Responsible for the development and implementation of Chemalysis' computerized sample tracking/management system. Skilled in dBASE software programming for laboratory automation and management.

1986 to 1988: Chemist, Biospherics Incorporated, Beltsville, MD.

Responsible for analyzing volatile organic samples by EPA methods 601 and 602. Extensive GC experience with purge and trap, FID/PID/HALL detectors and packed and capillary column analytical techniques.

Conducted all volatile organic analyses for a ground water study at a hazardous materials site on an industrial facility for 2 years. Analyzed hundreds of 601 and 602 samples on extremely rapid turnaround.

Skilled in personal computer applications for data transfer and reporting.

Training Courses

Hewlett-Packard RTE GC/MS Operating Course, July 1989

GREGORY ALLEN
Analytical Chemist

Education

B.A. Biology, Northeastern University, 1986

Experience

June, 1988 to present: Analytical Chemist, ChemAlysis Incorporated, Laurel, MD.

Responsible for analysis of pesticides, herbicides, and PCB's by EPA 600 and 8000 series and proprietary methods. Conducts pesticide residue analysis projects for major agricultural chemical producers. Develops and validates analytical methods, supervises extraction of plant and tissue samples, and performs analysis by GC. Extensive GC experience on organic analyses using FID and ECD detectors. Develops analytical schedules to ensure that all work is completed according to client requirements. Experience in operation of HP 5970 for the analysis of semivolatiles and pesticides.

1986 to 1988: Environmental Scientist, Biospherics Incorporated, Beltsville, MD.

Performed organic analysis of several thousand soil, water, and air samples for pesticides and PCB's. Extensive experience in wet preparation of samples for instrumental analysis. Conducted environmental sampling of numerous matrices including ground water, industrial effluent, transformer oils, and soils.

1984 to 1986: Laboratory Analyst, Organic Laboratory, ENSECO Inc., Cambridge, MA.

Coordinated analytical chemistry of bioaccumulation studies on marine invertebrates for government clients. Analyzed samples for pesticides, PCB's, and petroleum hydrocarbons using GC FID and ECD. Performed environmental sample collection, preparation, and extraction for semi-volatile organics.

Professional Affiliations

American Chemical Society
Phi Sigma Biological Honor Society

Training Courses

Hewlett-Packard RTE GC/MS Operator Course, July 1989

DAVID V. CABILES
Analytical Chemist

Education

B.S. Chemistry, University of Nueva Caceres, 1976
B.S. Ch.E., University of Nueva Caceres, 1974

Experience

October 1, 1989 to Present: Analytical Chemist, ChemAllysis, Incorporated, Laurel, Maryland.

Analyzes residues and metabolic products in various matrices by HPLC. Assists in scheduling work flow, sample management, and equipment maintenance. Prepares samples for instrumental analysis through standard wet laboratory techniques. Reduces raw data into report formats for submission to QA/QC. Supervises assigned technicians. Follows all GLP protocols.

January 12, 1989 to September 30, 1989: Extraction Technician, ChemAllysis, Incorporated, Laurel, Maryland.

Performs solvent extraction of soil, plant tissue, animal tissue, and water samples for instrumental analysis. Highly skilled in several extraction protocols. Extensive experience with both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

1986 to 1988: Night Auditor, Embassy Suites, Inc., Alexandria, Virginia.

Responsible for auditing customer's charges as well as hotel's transactions nightly. Balanced sales for each shift against the computer readout. Prepared and distributed nightly reports of all business transactions to sectional heads of department.

1981 to 1986: Analytical Chemist, Planters Product, Inc., Bataan, Philippines.

Performed approximately 90 laboratory analyses per day of various plant samples. Supervised and evaluated trainees in the analytical methods and techniques used. Wrote and submitted reports of chemical analyses and recommended appropriate method of application of fertilizer to ensure most economical use of raw materials. Monitored plant pollution levels. Prepared laboratory reagents and bench chemicals.

DONNA S. DAVIS
Analytical Chemist

Education

B.S. Chemistry, College of William & Mary, 1982

Experience

February, 1990 to Present: Analytical Chemist, ChemAlysis, Incorporated, Laurel, MD

Acts as project leader in the testing for pesticide residues, validates new analytical methodologies, plans sample throughput for projects, supervises technicians in sample preparation, monitors work status of projects, analyzes extracts by gas liquid chromatography, reduces data and generates reports, reports progress of studies to Laboratory Supervisor, maintains analytical equipment, responsible for maintaining sufficient laboratory supplies for studies, communicates with clients in regards to projects.

January, 1989 - January, 1990: Graduate Assistant, University of New Orleans

Position involved teaching the laboratory portion of introductory chemistry while pursuing a graduate degree and maintaining a grade point average of 4.0.

1986 to 1988: Chemist, Water and Waste Water Laboratory

Responsibilities included assuring compliance with EPA's Safe Drinking Water Act, as well as supervision of technicians performing chemical and microbial water quality tests. Conducted trihalomethane, pesticide, herbicide and PCB analysis by gas chromatography and heavy metal analysis using flame and furnace atomic absorption spectroscopy. Initiated a program to test for volatile organic carbons in drinking water by GC/MS resulting in the laboratory receiving EPA certification.

1985 to 1986: Chemist, Quality Assurance Laboratory

Primary duties involved Microtrac Particle Size Analyzer research directed at developing methods to determine particle sizes of solid propellants. Additional responsibilities included wet chemical analysis of raw materials to verify compliance with military specifications for use in station propellant production.

1982 to 1983: Chemist, Analytical Laboratory

Responsible for the operation, methods development and maintenance of data controlled gas and liquid chromatographs. Specific experience with chromatographic separation of carbohydrates, fatty acids and amino acids. Promoted to Laboratory Supervisor January 1984.

GEOFF D. MALONEY
Analytical Chemist

Education

B.S. Biochemistry, University of Maryland, 1987.

Experience

September 1, 1989 to Present: Analytical Chemist, ChemAllysis, Incorporated, Laurel, Maryland.

Analyzes residues and metabolic products in various matrices by GC. Experienced in use of FPD, NPD and ECD detectors. Assists in scheduling work flow, sample management, and equipment maintenance. Prepares samples for instrumental analysis through standard wet laboratory techniques. Reduces raw data in report formats for submission to QA/QC. Follows all GLP protocols.

August 29, 1988 to August 31, 1989: Extraction Technician, ChemAllysis, Incorporated, Laurel, Maryland.

Performs solvent extraction of soil, plant tissue, animal tissue, and water samples for instrumental analysis. Highly skilled in several extraction protocols. Extensive experience with both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

1984 to 1988: Laboratory Technician, U.S. Department of Agriculture, Beltsville Human Nutrition Research Center, Beltsville, Maryland.

Laboratory technician in Vitamin and Mineral Laboratory, specializing in selenium research. Work involved use of HPLC and Atomic Absorption Spectrophotometer to determine vitamin levels in biological samples, column chromatography of muscle proteins, determination of enzyme activity. Participated in and helped conduct human studies. Prepared animal tissues for analysis.

CATHERINE J. REITER
Analytical Chemist

Education

B.A. Biology and Environmental Sciences, University of Virginia,
1989

Experience

October 1, 1989 to Present: Analytical Chemist, ChemAlysis,
Incorporated, Laurel, Maryland.

Analyzes residues and metabolic products in various matrices by GC. Assists in scheduling work flow, sample management, and equipment maintenance. Prepares samples for instrumental analysis through standard wet laboratory techniques. Reduces raw data in report formats for submission to QA/QC. Follows all GLP protocols.

June 12, 1989 to September 30, 1989: Extraction Technician,
ChemAlysis, Incorporated, Laurel, Maryland.

Performs solvent extraction of soil, plant tissue and water samples for instrumental analysis. Highly skilled in several extraction protocols. Extensive experience with both partition and column clean-up. Follows ChemAlysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

ANDREA RIES
Analytical Chemist

Education

B.S. Chemistry, University of Maryland, College Park, 1989
University of Baltimore School of Law, August 1989 - Present

Experience

September 1989 to Present: Analytical Chemist, ChemAlysis, Incorporated, Laurel, Maryland.

Analyzes water and soil for trace metals by flame and graphite furnace atomic absorption spectrophotometry, and mercury by cold vapor spectrophotometry. Oversees preparation and analysis of Cyanides, Phenols and other inorganic parameters by UV/VIS spectrophotometry and oil/grease and total petroleum hydrocarbons by infrared spectrophotometry. Manages computer databases of analytical data to generate reports using commercial spreadsheets including Lotus 1-2-3. Maintains Quality Control Charts/Graphs using computer database.

January 1989 - September 1989: Inorganic Technician, ChemAlysis Incorporated, Laurel, Maryland.

Prepared water and soil samples for trace metal analysis. Prepared and analyzed samples for Cyanide, Phenols and other inorganic parameters by UV/VIS spectrophotometry and other wet chemistry techniques. Extracted soil for pesticide residue analysis. Entered data and assisted with report generation using commercial computer spreadsheets (Lotus 1-2-3).

September, 1988 to December, 1988: Administrative/Research Assistant, National Public Services Research Institute, Landover, Maryland.

Assisted with library search of technical journals. Performed data entry and manipulation using commercial spreadsheets including Lotus 123.

May, 1988 to September 1988: Laboratory Intern, W.R. Grace & Co., Washington Research Center, Columbia, Maryland.

Conducted small-scale laboratory experiments and prepared samples for reactions and analyses.

JOYCE JOHNSON
Senior Extraction Technician

Experience

January 1, 1988 to Present: Senior Extraction Technician, ChemAlysis Incorporated, Laurel, MD.

Prepares solvent extraction of soil, plant tissue, animal tissue, and water samples for instrumental analysis. Over 8 years experience performing thousands of residue extractions for numerous pesticide projects. Well versed in a variety of extraction protocols and methodologies. Follows ChemAlysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

March 1, 1981 to December 1987: Chemistry Technician, Tegeris Laboratories, Temple Hills, MD.

Sample control officer for pesticide residue programs. Extracted analytical samples from different matrices, performed extract clean-up by solvent partitioning, and prepared reagents and standards for analysis. Assisted animal technicians in administering anesthetic agents, obtaining body weights, observing pharmacotoxic signs, and performing necropsies in toxicology studies.

May 1, 1978 to February 1981: Stock Room/Shipping and Receiving Technician, Pharmacopathics Research Laboratories, Laurel, MD.

LEELA A. MATHAI
Senior Extraction Technician

Education

B.C.M. College, Kottayam, India
B.S.C., Biology Chemistry

Buchanan Institution, Kattayam, India

P.G. Community College, Largo, MD
Lab Tech

Experience

July 1988 to present: Extraction Technician, ChemAllysis, Incorporated, Laurel, MD

Performs solvent extraction of soil, plant tissue, animal tissue and water samples for instrumental analysis. Well versed in a variety of extraction protocols and methodologies. Extensive experience with both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

January 1988 to June 1988: Study Coordinator/Junior Toxicologist, Tegeris Laboratories, Inc., Temple Hills, MD

Duties included: Responsibility under supervision by the Study Director for the implementation of specific toxicological research projects in accordance with the approved protocol, Standard Operating Procedures and Good Laboratory Practice Regulations (GLP) and accordingly to assist other Study Coordinators in the implementation of other approved projects. These duties more specifically include the following activities: Receiving animals, all phases of maintaining and caring of animals, observing animals during quarantine period for abnormalities and/or signs of toxicity, randomizing animals, ear punching animals for identification purposes, mixing feed with an exact amount of compound, administering test substances to laboratory animals via gavage, data collecting including body weight and feed consumption performing statistical tests on raw data (body weight, feed consumption, etc.) observing animals for clinical, pharmacological and toxicological signs, collection of metabolic samples of blood, urine and feces, monitoring both temperature and humidity throughout the laboratory, maintaining cleanliness of laboratory and animal quarters, necropsy of dead and sacrificed animals, blocking of animal tissues for histology, surgical removal of mammary tumors, record keeping, report generation and proofing.

1987: Lab Technician, E.S.S. Laboratory Service, 5111 College Avenue, College Park, MD

Duties included: Plating samples counting calories, test freeze point.

1977 to 1980: Lab Technician, Nairs Hospital and Clinic, Quilon, Kerala, India

RUTH RAMSEUR
Senior Extraction Technician

Education

Medical Laboratory Technology, Prince George's County Community College, 1976

Experience

January 1, 1988 to Present: Senior Extraction Technician, ChemAllysis Incorporated, Laurel, MD.

Performs extractions of soil, plant tissue, animal tissue, and water samples for analysis by GC or HPLC. Has over 10 years of experience in residue extractions working on extensive pesticide projects for some of the largest international chemical manufacturers. Knowledgeable about GLP requirements and ChemAllysis' SOPs.

April 1, 1979 to December 30, 1987: Chemistry Technician, Tegeris Laboratories, Laurel, MD.

Responsible for the preparation of samples for pesticide residue studies. Weighed and prepared reagents and standards. Prepared suspensions and sterilized equipment for instillations. Assisted animal technicians in administering anesthetic agents, obtaining body weights, observing pharmacotoxic signs, and performing necropsies in toxicology studies.

1966 to 1977: Recovery Room Technician, Children's Hospital National Medical Center, Washington, D.C.

VIOLETA BURGOS
Extraction Technician

Education

B.S. Biology, University of Puerto Rico, Cayey, Puerto Rico

Professional Affiliations

Puertorican Microbiology Society

Experience

July, 1989 to Present: Extraction Technician, ChemAllysis Incorporated, Laurel, MD.

Performs solvent extraction of soil, plant tissue, animal tissue and water samples for instrumental analysis. Well versed in a variety of extraction protocols and methodologies. Extensive experience with both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

1982 to 1985: Biology Research Technician, University of Puerto Rico, Cayey, Puerto Rico.

Assisted in the development of all research procedures to the department staff. Prepared reactives and biology laboratory lectures. Prepared and gave laboratory examinations.

1985 to 1986: Drug Clerk, Peoples Drug, Manassas, Virginia

Responsible for making requisitions for prescription drugs. In charge of receiving vendors.

CHRIS HERNANDEZ
Extraction Technician

Education

B.S. Chemical Engineering, Adamson University, Manila
Philippines, 1983

Basic Computer Programming, Ateneo de Manila University,
Manila, Philippines

General Science, Montgomery College, Takoma Park, Maryland

Experience

August 1990 - Present: Extraction Technician, ChemAllysis
Incorporated, Laurel, MD

Performs solvent extraction of soil, plant tissue, animal tissue
and water samples for instrumental analysis. Well versed in a
variety of extraction protocols and methodologies. Extensive
experience with both partition and column clean-up. Follows
ChemAllysis' internal SOPs to ensure compliance with all GLPs and
other Federal guidelines.

May 1989 to Present: Vialing Technician, Life Technologies,
Gaithersburg, MD

Handling bulk products in the manner that is required. Receiving
bulks and writing up worksheets, maintaining department records
and assorted paper works in an accurate and free fashion.
Operates vialing department machines i.e., Laser, Josco, Avery,
APS, etc.

Crossed Training to Quality Control Department, doing volume
checking, enzymatic assay test using gel electrophoresis. Bio-
organic Laboratory, preparing react buffers, agarose and
preparing components for photogene kit.

June 1984 to May 1989: Nursing Home Supervisor, Medlantic Manor
at Layhill, Silver Spring, MD and Sylvan Manor Nursing Home,
Silver Spring, MD

March 1983 to June 1984: College Instructor, Trinity College,
Manila, Philippines

Taught Algebra, Trigonometry, Chemistry and Basic Computer
subjects. Conducted major examinations and quizzes. Prepared
lesson plans, records and computed student grades.

Professional Affiliations

Adamson University Chemical Engineering Society
Chemical Engineering Society of Southern California

EMILY S. LANDIS
Extraction Technician

Education

Ph.D. Geology, University of Maryland, in progress
M.S. Geology, University of Toledo, 1981
A.B. Geology, Miami University, 1989

Experience

January 1990 - Present: Extraction Technician, ChemAlysis Incorporated, Laurel, MD

Prepares soil and water for trace metal analysis using EPA guidelines. Proficient in titrimetric and colormetric techniques. Performs organic extractions for priority pollutant analysis.

1989 - 1990: Electron Microscopist, Aerosol Monitoring Analysis, Analytical Lab, Lanham, MD

Performed special research projects related to sample preparation for transmitted electron microscopy and laboratory accreditation.

1986 - 1989: Physical Scientist, National Institute of Standards and Technology (formerly Bureau of Standards), Center for Analytical Chemistry, Microanalysis Group, Gaithersburg, MD

Specialized in developing and characterizing techniques for submicrometer materials analysis by light and electron microscopy for asbestos standards.

1985 - 1986: Museum Technician, Smithsonian Institution, Washington, DC, Scientific Event Alert Network of the Global Volcanism Program, Dept. of Mineral Sciences

1983 - 1985: Graduate Teaching Assistant, Geology Dept., University of Maryland, College Park, MD

Taught laboratory courses for Introductory Geology and Optical Mineralogy.

1982 - 1983: Curator's Assistant, Colburn Memorial Mineral Museum, Asheville, NC

Identified and catalogued mineral and fossil specimens.

Professional Affiliations

Mineralogical Society of America
American Geophysical Union
Geological Society of Washington
Microbeam Analysis Society

Publications

Nielson, R.L., Landis, E.S., Ceci, V.M., and Postonc., The
Commingling of Diverse Magma Types in the Flagstaff Lake Igneous
Complex (invited paper), Jackson Memorial Volume of the Maine
Geological Survey Bulletin, Vol. 2 - Igneous and Metamorphic
Petrology of Maine (in press).

In preparation: Landis, E.S., Nielson, R.L., Baker, D.R.,
Evidence for a Magmatic Origin of Almandine Garnet in Gabbros of
the Flagstaff Lake Complex, West-Central Maine.

DONALD L. SHELLY JR.
Extraction Technician

Education

B.S. Forestry and Wildlife Management, Virginia Polytechnic Institute and State University, May 1989

Certified Laboratory Animal Technician

Experience

August, 1989 to Present: Extraction Technician, ChemAllysis Incorporated, Laurel, MD

Performs solvent extraction of soil, plant tissue, animal tissue and water samples for instrumental analysis. Well versed in a variety of extraction protocols and methodologies. Extensive experience with both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

1988 - 1989: Tree & Shrub Specialist, ChemLawn Services Corporation, Landover, MD

Responsibilities included: sales; resolving customer complaints; applying pesticides and fertilizers; providing advice on proper horticultural practices.

1985 - 1988: Laboratory Animal Technician, Office of Animal Resources, Virginia Polytechnic Institute and State University, Blacksburg, VA

Responsibilities included: maintaining animal health; performing scientific experiments; performing surgical procedures; providing technical advice.

1988 - 1988: Data Entry Technician, Department of Fisheries and Wildlife Science, Virginia Polytechnic Institute and State University, Blacksburg, VA

Responsible for entering research data into computer files.

1981 - 1982: Dining Room Supervisor, Williamsburg Lodge Hotel, Williamsburg, VA

Responsible for supervising employees and enforcing quality control.

MATTHEW A. WARE
Extraction Technician

Education

B.S. Political Science, Norfolk State University, Norfolk, VA.

Experience

December, 1989 to Present: Extraction Technician, ChemAllysis, Incorporated, Laurel, MD.

Performs solvent extraction of soil, plant tissue, animal tissue, and water samples for analysis by GC or HPLC. Well versed in a variety of extraction protocols. Skilled in both partition and column clean-up. Follows ChemAllysis' internal SOPs to ensure compliance with all GLPs and other Federal guidelines.

June, 1988 to December, 1989: Sample Receiving Clerk, ChemAllysis, Incorporated, Laurel, MD.

1983 to 1987: Student Aide - Library Assistant, Norfolk State University, Norfolk, VA.

1987 to 1987: Outreach Counselor, YMCA of Tidewater, Norfolk, VA.

1986 to 1986: Shift Supervisor, Reliance Security Services, Norfolk, VA.

1984 to 1987: Customer Service, Toys "R" Us, Norfolk, VA.

1979 to 1981: Mail Clerk, Printer, City Hall Printing Bureau, Lynchburg, VA.

APPENDIX D
Chronic Aqueous and Solid-Phase Toxicity Testing
Biological Monitoring, Inc.

AR302251

BIOLOGICAL MONITORING, INC.
QUALITY ASSURANCE/QUALITY CONTROL MANUAL

Version 3.0

Prepared by:
Biological Monitoring, Inc.
P.O. Box 184
Blacksburg, Virginia 24063
703-953-2821

September 8, 1989

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Biological Monitoring, Inc., Blacksburg, Virginia

AR302252

SUMMARY OF QUALITY ASSURANCE PROCEDURES

As in all studies of this nature, a Project Manager is assigned to this study upon authorization for BMI to initiate work. The Project Manager is generally a Ph.D. scientist with sufficient experience and background to see that the study is performed accurately and successfully. The Project Manager appoints Project Scientists who are responsible for carrying out the study of design. Project Scientists at BMI are audited quarterly to assure that they are trained and knowledgeable in toxicity test methods.

All bench sheets, including sample chain-of-custody sheets are included in the final report. In addition, reference toxicant data for each species tested are included in the report to assure that the test organisms were in good health at the time of testing. Furthermore, since all species in this study are cultured at BMI, we have daily culture log sheets which document the health and maintenance of the test organisms used.

Finally, all chemical and biological analyses are subject to BMI's Quality Assurance Program, conforming to our written Quality Assurance Manual which is available upon request.

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BIOLOGICAL MONITORING, INC.
Quality Assurance/Quality Control Manual

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1.0 PURPOSE

The purpose of this document is to define the Good Laboratory Practices (GLP) as followed by Biological Monitoring, Inc. (BMI). GLP's are concerned with the organizational process and the conditions under which laboratory studies are planned, performed, monitored, recorded, and reported at BMI. GLP's essentially consist of two components: Quality Assurance (QA) and Quality Control (QC) programs. The objective of the QA program is to establish BMI's ability to produce consistent, valid data. Documentation is the key element of this program. The QC program is a statement of BMI's intention to provide good, valid data and its specific actions taken to implement that task.

2.0 ORGANIZATION OF PROGRAM

BMI's management has an ongoing commitment to a high quality assurance of data collected and/or received and interpreted by the staff. This commitment is expressed by careful organization and planning and continuous training and inspection of all staff members at BMI.

2.1 Laboratory Manager

The Laboratory Manager is supervisor for all technical personnel. The minimum requirements for this position are a bachelor of science degree from an accredited college or university in a biological science and at least three years laboratory experience in aquatic toxicity testing, or a master of science degree in a biological science and at least one year of laboratory experience in aquatic toxicity testing. The person in this position is ultimately responsible for the overall performance of the laboratory in its execution and reporting of analyses. Only the Laboratory Manager can make changes in laboratory procedure. Finally, the Laboratory Manager may serve as Quality Assurance Officer for any given project on which he is not directly technically involved, or appoint a qualified person for this position.

2.2 Quality Assurance Officer

On any given project, including laboratory and field work, a Quality Assurance Officer (QAO) is assigned. This may be the Laboratory Manager or one of the senior members of the staff who, as proven by performance reviews and audits, has the required experience and expertise to supervise the project. The QAO, though not technically involved with specific projects, performs the following duties for a given project:

- a. Coordinates, supervises, and inspects all methods and procedures for a project.
- b. Inspects staff qualifications to insure the preparedness and training of Project Scientists and technicians.
- c. Evaluates and reviews all data and reports resulting from projects prior to reporting or transmitting data to permanent storage.
- d. Appoints a Quality Control Officer for each project.

2.3 Quality Control Officer

The Quality Control Officer (QCO) assigned to a given project is responsible for the accurate collection of data and sees to it that Standard Operating Procedures (SOP's) and GLP's are carried out. The QCO is considered synonymous with Project Manager. The specific duties of the QCO, or Project

Manager, are as follows:

- a. Inspect and make sure SOP's and GLP's are executed correctly by the staff.
- b. Inspect and make sure all protocols and methods agreed upon in advance by the QAO are carried out for the project.
- c. Report to the QOA any departures, problems, or inconsistencies either in data collected or methods utilized.
- d. Monitor performance of the laboratory staff, check that data is logged in appropriately, and check performance of tests.
- e. Approve all laboratory data before the data is given to the QAO.
- f. Insure that all equipment, reagents, and other laboratory supplies meet specified criteria contained in this document.

2.4 Project Scientist

The appointment of a Project Scientist for each field or laboratory project is handled by the Laboratory Manager or someone in a superior position. Only persons qualified by successfully completing technical audits may serve as Project Scientist whose duties include making sure appropriate equipment (i.e. glassware, meters and other instruments, chemicals, field equipment, and specific apparatus unique to any given project) are available and in proper working condition at the start of a project. Also, availability of test organisms, access to proper dilution water, and supplies for special feeding requirements of test organisms (when necessary) are the responsibility of the Project Scientist.

2.5 Laboratory Staff Training

All laboratory staff are first trained for a minimum of several weeks prior to performing routine field work and laboratory procedures and tests independently. During this training period, staff members work with a senior member who checks all aspects of their performance. Once the laboratory trainer is convinced that all SOP's and GLP's are being followed by the trainee, the trainee begins to perform some aspects of the procedures independently. Checks are made of their performance continuously for a minimum of 3 - 6 months. Only after an extensive audit are staff members permitted to perform test procedures under the supervision of the QAO alone. Once initial training has been completed for a given task/test the trainee is subject to a performance inspection audit. The audit is performed by a senior staff member and includes checks of all aspects of the task/test. Two consecutive audits must be passed before the staff member is allowed to perform the task/test independently. Failure to pass an audit results in review of the problems found, repeating the training procedure with closer supervision and instruction, and two follow-up laboratory testing performance inspections.

2.6 Laboratory Staff Performance Inspection

After successfully completing the training requirements as stated in section 2.5, a technical staff member is formally audited on personal performance on a quarterly basis. Any questionable performance by any staff member at any time is given immediate attention by the Laboratory Manager. The staff member who does not successfully pass an audit is not allowed to perform testing procedures and must be retrained under the supervision of the Laboratory Manager.

2.7 Aquatic Biologist Certification

Currently few state or federal agencies have certification programs for biological testing laboratories. The agencies which do have programs certify the entire laboratory rather than the staff performing the testing. BMI feels that technical staff performance is a key aspect of generating accurate data. Therefore, BMI has implemented an in-house Aquatic Biologist certification program. Following successful completion of appropriate training and performance inspection audits, technical staff achieve Aquatic Biologist Certification.

There are currently three levels of certification: Aquatic Biologist I, II, and III. The required training and successful completion performance inspection audits for each level are described below:

Aquatic Biologist I - routine laboratory maintenance

- routine physiochemical analysis (dissolved oxygen, pH, conductivity, temperature, alkalinity, hardness, and chlorine)
- test organism culturing and maintenance
- acute toxicity testing (static and static renewal)

Aquatic Biologist II - Aquatic Biologist I certification

- chronic toxicity testing (static-renewal)
- acute flow-through testing
- test data analysis and processing

Aquatic Biologist III - Aquatic Biologist I and II certification

- chronic flow-through testing
- aquatic field studies

3.0 LABORATORY FACILITIES

3.1 Laboratory Space and Arrangement

BMI's laboratories include a fixed laboratory facility with 1800 square feet of available floor space, 127.5 feet of total linear bench spaced and a mobile laboratory facility with 176 square feet. Both hot and cold water are available as well as dechlorinated water. The space is arranged so that different functional processes (i.e., culturing, washing, wet-lab, toxicity testing, etc.) are separated from each other. This helps to insure safety and the satisfactory maintenance of each station within the laboratory.

3.2 Environment

Air temperature is maintained at $20^{\circ}\pm 2^{\circ}\text{C}$ year-round in the laboratory via a central heating system, air conditioners, and thermostats. Maximum and minimum temperatures for the laboratory are observed and recorded daily to insure that proper air temperature is maintained. Appropriate corrective action is taken if the temperature is less than 18°C or greater than 22°C .

The photoperiod (which is 16 hours of daylight and 8 hours of darkness) is regulated and maintained via two centralized automatic timer systems. The accuracy of the of the photoperiod is checked quarterly to insure correct performance of the timers. Records are kept of these checks including any necessary corrective actions taken. These records are kept in the permanent archives.

Aeration for cultures and testing purposes is provided through a centralized oilless air compressor system. Quarterly checks of compressor functioning and air valves are made and recorded. In addition, daily checks of the air flow in cultures also insures quick corrective action in the event of a failure in the air system. Backup aeration systems are available.

The main source of water used for culturing and testing purposes is dechlorinated Blacksburg municipal water. Water is dechlorinated with a Culligan model #446468 dechlorinator which uses activated carbon and a 0.2 micron filter. This instrument is back-flushed automatically every fourth day. All dechlorinated water is checked for total residual chlorine using an amperometric titrator (Standard Methods) before use and this information is recorded on the Dechlorinated Tapwater and Dechlorinator Log Sheet. Quarterly inspections of the log sheet are made by the Quality Assurance Officer and a record of the inspections is kept in the permanent archives. Hardness, alkalinity, and pH of the dechlorinated water are measured and recorded at least weekly. Mean and standard deviation values are calculated for these parameters on a quarterly basis. Semi-annually, samples of dechlorinated water are tested by an outside chemical laboratory for the presence of EPA

priority pollutant organics and metals.

Dechlorinated water used for culture purposes and toxicity testing is first aerated vigorously for at least 48 hours. Total residual chlorine is measured on all batches of dechlorinated water prior to use. Water is not used if chlorine is detected (detection limit = 0.01 mg/L). Water is dispensed into 55 gallon polyethylene containers designated for dechlorinated water storage only. The water is then aerated continuously. Log sheets are kept for recording all information concerning dechlorinated water storage and this information is kept on permanent file.

4.0 LABORATORY TESTING EQUIPMENT

4.1 General

Instruments used for routine measurements of chemical and physical parameters such as pH, DO, temperature, conductivity, salinity, etc., are calibrated and standardized according to instrument manufacturer's instructions. Statistical limits have been established for analysis parameters and appropriate corrective action is taken if these limits are exceeded. Corrective actions include notifying the QAO of the problem, eliminating the possibility of technical error, and servicing the instrument if necessary. Records are kept on log sheets of calibration, standardization and service required, and the log sheets are kept on file. For Standard Operating Procedures for individual instruments see BMI's SOP Document, Section B.0.

4.2 Balances

Balances are calibrated quarterly using standard weights. Results of the calibration procedure and any necessary adjustments made are recorded.

4.3 Dilutor

The flow-through proportional dilutors used at BMI are manufactured by Specialized Environmental Equipment Company. All surfaces that come in contact with the test waters are glass, teflon, or type 316 stainless steel.

Effluent or test compound and diluent enter proportioning head boxes through electronically controlled solenoid valves. When the proportioning chambers are filled, an electric high-level probe activates the proportioning chamber solenoids to drain the chambers. Simultaneously, the fill solenoids are closed. The test waters flow by gravity through adjustable standpipes from the proportioning boxes into stainless steel mixing cups, where effluent and diluent are mixed. The mixture then flows by gravity through two separate lengths of tubing to the exposure chambers. An electric low-level probe in the proportioning chambers activates a time delay relay which restarts the fill cycle after a set interval. The dilutors can be set to do ten complete water volumes per day, or a 90% turnover time of approximately 5 hours. The test concentrations delivered by the dilutor are calibrated prior and during all toxicity tests.

4.4 Water Quality Meters

All meters used to test water quality for laboratory and field work purposes are calibrated prior to use using known standards and meter manufacturer's

instructions. Calibration and standardization results are recorded as are any corrective actions taken when meter calibration readings do not fall within acceptable limits set by the manufacturer. Directions for calibration and corrective actions are found in manufacturer's manuals which are kept on file in the laboratory.

4.5 Water Baths and Refrigerators

Several closed tanks are available for use as water baths. The availability of several types of water heaters and chiller units makes possible water baths ranging from near freezing (2°C) to boiling (100°C). Water bath temperatures are checked with calibrated thermometers and continuous temperature recorders.

4.6 Reagents

All reagents received by BMI are logged in with the following information: date of delivery, expiration date, and where the reagent was obtained. All reagents are stored in designated areas away from other facilities at BMI. Expiration dates of reagents are checked monthly and expired chemicals are discarded. A record is kept of the reagents in stock, and their expiration dates.

4.7 Glassware

All glassware (including aquaria) is stored separately from other materials and supplies. Glassware is washed as per EPA guidelines. (See Standard Operating Procedures Document A.2.) Quarterly checks of glassware are made by the Quality Assurance Officer and these checks are recorded in a log in the archives. Corrective action includes a review of glassware cleaning techniques and a check on cleaning equipment (dishwasher, water heater) involved.

5.0 SAMPLE AND EFFLUENT HANDLING

5.1 Sample Collection and Shipment

All samples received by BMI must be accompanied by a Sample Collection and Chain of Custody Form which must be completed by the sampler and includes the client's name, name of the sampler, NPDES permit number, description of sampling site and collection methods, date and time of collection, method of sample shipment, and type of testing required. This form acts as a permanent record for the sample and a copy is included with the analysis report.

If sampling is to be performed by the client, sampling instructions and appropriate sample containers and coolers are provided by BMI. Sample containers may be reusable glass or new disposable polyethylene containers.

Samples are shipped at 4°C and testing must be initiated within 36 hours of sample collection. Therefore, samples are shipped via overnight delivery service or hand delivered to BMI.

5.2 Handling of Samples Received for Laboratory Testing

Upon arrival at BMI, all samples are promptly processed by trained laboratory personnel. All samples are given unique identification numbers when they arrive. The date and time of receipt is recorded in addition to sample temperature, pH, conductivity, salinity, and total residual chlorine (TRC). Sample collection information is reviewed and the sampler is contacted if clarification is needed.

If test initiation procedures are not scheduled to begin upon receipt of the sample, it is stored at 4°C. If testing is scheduled to initiate upon arrival of the sample (which is usually the case), the Laboratory Manager or his designated representative reviews the sample information and scheduled testing, and assigns a project scientist. A laboratory work order describing the test(s) to be performed and specific test conditions is issued to the project scientist and testing is initiated.

Upon the initiation of a project and during the course of testing, information regarding the project is recorded on the Project Progress Log Sheet by the Project Scientist. Information recorded includes client's name, project description, sample name and I.D. number, project scientist, QCO, date testing is complete, person preparing report, person reviewing report, and date report is mailed out. This serves as a concise record of the testing being performed on each sample and the progress of each project.

5.3 Invalid Samples

Should the analyst discover any abnormalities in sample collection or handling such as improper field preservation of a sample, or improper shipment of a sample, the Laboratory Manager is alerted. In most cases the client will be notified and a resampling requested.

5.4 Samples for Chemical Testing

Chemical sampling is currently sub-contracted by BMI. The Laboratory Manager is responsible for making arrangements for scheduling, sampling, and shipping should chemical testing be requested by a client.

6.0 ORGANISM CULTURE AND MAINTENANCE

6.1 Record Keeping

All organisms, either cultured in the BMI laboratory or obtained from an outside source, are carefully maintained by staff Aquatic Biologists who keep detailed records of all culture procedures via the use of log sheets. These sheets contain pertinent data concerning the maintenance of each particular culture and the initials of the technician responsible for maintenance. At the minimum, weekly checks of the log sheets are made by the Quality Control Officer to insure the proper recording of data on log sheets. A culture audit is completed by the QCO quarterly or more often as necessary. All log sheets are kept on permanent file and copies of relevant log sheets are included in an analysis report.

6.2 Culture Systems Maintenance

All cultures are under a stringent maintenance schedule which includes periodic cleansing of tanks, frequent checks of the water quality, and the health and well-being of the organisms. When a tank is cleaned or in some other way handled, this information is recorded on the culture log sheet and initialed by the person who performed the maintenance.

Organisms used to stock cultures are obtained from reputable sources with established quality assurance. Information regarding the source of culture organisms and the date they were obtained is kept on file to assure a means of tracking the approximate age of the organisms and to aid in quality control.

Organisms are rotated and/or replaced on a schedule designed to keep availability of healthy test organisms at a maximum. Detailed records are kept of culture systems maintenance by qualified staff members and the Quality Control Officer.

6.2.1 Algae

Currently, Ankistrodesmus falcatus, Chlamydomonas reinhardt, and Selenastrum capricornutum are continuously cultured in the growth chamber at BMI. The procedure for culturing these species is explained in Section E.0 of BMI's Standard Operating Procedures (SOP) Document.

6.2.2 Invertebrates

The following invertebrate organisms are currently cultured and/or maintained in BMI's laboratory facility: Daphnia pulex, Daphnia magna, Ceriodaphnia

dubia, Mysidopsis bahia, Artemia sp., and aquatic insect larvae (Heptageniids and Siphonurid mayflies). The SOP's for the culture and maintenance of these organisms are included in the SOP Document (Section D.0). Other invertebrates which have been cultured at BMI include various amphipod species, crayfish, marine and freshwater bivalves, and grass shrimp (Palaemonetes pugio).

6.2.3 Fish

The following fish species are cultured and/or periodically maintained at BMI: Pimephales promelas (fathead minnow), Fundulus heteroclitus (killifish), Morone saxatilis (striped bass), Salmo gairdneri (rainbow trout), Salmo trutta (brown trout), and Cyprinodon variegatus (sheepshead minnow). The SOP's for the culture and maintenance of fish are included in BMI's SOP Document in Section C.0.

6.2.4 Outside Suppliers

If fish need to be obtained from an outside supplier, special quality assurance procedures are instituted to insure the health of the organisms obtained and to reduce the risk of contamination of in-house cultures.

First, organisms are obtained from reputable suppliers who guarantee the health of their cultures and the way in which the organisms are packed and transported.

Second, communication is established with the supplier, prior to shipment, to insure professional handling of the organisms and to insure adequate preparation time to receive the organisms into the laboratory. These communications are also used to determine the specific physiochemical conditions under which the organisms were maintained by the supplier. This information is used to make any necessary adjustments in the conditions of the receiving tanks to minimize stress resulting from the organism transfer process.

Third, organisms received from outside suppliers are quarantined for at least 1 week prior to testing or introduction into the main culture area. If necessary, fish are given prophylactic treatment with a commercially prepared fungicide and bactericide as per the manufacturer's recommendations. These practices are intended to reduce the risk of disease transfer to other in-house cultures and to maintain the health and well-being of the newly obtained culture.

A record of receipt of fish obtained from an outside supplier is recorded in the Fish Log File which contains the following information: Name and address of supplier, fish species, date received, and a batch number. For specific

- details concerning receiving fish or other organisms from outside suppliers, refer to BMI's SOP Document, Sections C.4 and D.9.

6.2.5 Quality Control

In addition to the above culture maintenance protocols, BMI laboratory personnel are required to complete the Laboratory Procedure Checklist. This check sheet helps to insure that culturing techniques are carried out with sufficient frequency. The completed check sheet for each month is reviewed by the QAO and signed off if approved. Daily inspections are made by one of the QCO's to make sure that daily procedures are in fact carried out and that these procedures are done in accordance with BMI's SOP's and GLP's.

7.0 FIELD STUDIES

7.1 General

The particular nature of field sampling depends on the objectives of the study and habitat-specific characteristics of the field site. However, regardless of the specific type of field research, all involve some basic quality control features to insure the accurate collection of data. These features include selection of appropriate sampling sites, the frequency of sampling, the selection of sampling methods, calibration and maintenance of all sampling equipment, and correct preservation of samples, if necessary. A QA/QC plan is developed for each field project which includes a project manager and is designed to cover all areas of quality assurance and quality control requirements unique to each project.

7.1.1 Selection of Sampling Sites

Sampling sites in the field are chosen so as to be as unbiased as possible and to fulfill the study objectives. Replicates within a site are taken in order to sample representative areas and to determine the intrasite variability in the response variables. The following criteria should be taken into consideration when selecting the sampling site:

- a. Familiarity with historical data including biological, chemical, and physical nature of the site.
- b. Good definition of the study objective.
- c. Degree of accessibility.
- d. Whether or not the stands (or stations) appear to be representative.
- e. Availability or satisfactory adjacent stands, since it is convenient to establish more than one station at each field locality.
- f. Organism-specific: For fish, samples are taken in obscure and unlikely areas as well as at obvious locations; samples are made at all depths, not just surface and bottom. For other organisms, samples are taken to suit the special requirements.
- g. Habitat-specific: In rivers, samples are made upstream and downstream from the pollution source. In lakes, reservoirs, and other standing-water bodies where the zones of pollution may be arranged concentrically, stations are located in an area adjacent to the waste outfall and in an unaffected area.
- h. For aquatic vegetation: Three criteria are applied in the decision to include or reject a particular site:
 1. Following an initial survey, samples are prescribed in proportion to the area covered in each existing emergent vegetation type,
 2. Sampling at different water depths, and
 3. At least one emergent species has to be present.

If the objective of a study is qualitative in nature (to describe the flora and fauna of an area with a high degree of accuracy), a relatively large number of samples are collected from a large number of habitat types.

In order to choose appropriate sampling sites, experience and familiarity with the general study objectives and the site-specific characteristics are crucial. Well-qualified, experienced personnel oversee all site-selection in the field for this reason.

7.1.2 Frequency of Sampling

Frequency of sampling is of critical importance. In a sampling program, frequency influences the validity of data, particularly the precision and accuracy of the data. In general, the more frequent the sampling, the more precise and accurate the data.

There are many elements that determine the frequency of sampling. Among these elements some important ones are:

- a. The objective of the study.
- b. The organisms being studied.
- c. The availability of manpower.
- d. The availability of historical information.
- e. The limitation of time.
- f. The limitation of money.
- g. The adequacy of sampling equipment.
- h. Environmental factors.

Prior to the beginning of the sampling, study objectives must be defined clearly and carefully. For example, the frequency of sampling may vary from hourly, for a detailed study of diel variability, to quarterly (every third month), for a general estimation of seasonal variations, depending on the objectives. If the objective of the study is quantitative in nature, the increase in the frequency of sampling may increase the precision of the data, e.g., the estimation of fish populations in a body of water. Frequent samplings are also necessary in a pollutional study if the characteristics of effluents change or if spills occur.

Available manpower, time and money always determine the scope of study and the frequency of sampling, too. The sampling frequency is adjusted to limitations in personnel, time and money.

If sampling can be automated, more frequent samplings can be made than are otherwise possible. For example, automatic monitors can sample air, water or other media continually, e.g., hourly through the day, month, and year.

Biological factors such as organisms being studied determine the frequency of

sampling. That is, the frequency of sampling for plankton may differ from that for fish or other organisms because each studied organism has its unique biology, e.g., habitat types, and natural variability.

The frequency of sampling is sometimes determined by the available historical information attainable by searching literature (or work) by previous investigators.

Environmental factors also influence the determination of sampling frequency. For instance, sudden meteorological changes such as a hurricane storm may force biologists to sample more frequently in its aftermath.

7.1.3 Selection of Sampling Methods

Field sampling is performed as described in Weber (1973). For quantitative macroinvertebrate sampling, quantitative samplers (Surber - Hester - Dendy) are used. Nets and grab devices are used for qualitative samples. Care is taken to standardize all sampling techniques to insure replicate sampling effort at all sites. (For specifics on collection of Stenonema modestum (Mayflies) see SOP D.12 of the SOP Document.

7.1.4 Calibration and Maintenance of Equipment

Field equipment is calibrated (where appropriate) according to manufacturer's instructions prior to use. Should calibrations show unacceptable results, equipment is repaired or adjusted according to user's guides from manufacturers before sampling occurs.

Regular maintenance work consists of good, thorough cleaning after use, drying before storage, and proper storage. This applies also to the mobile laboratory facility.

7.1.5 Biological Sample Preservation

Upon obtaining a valid and representative sample in the field, sample preservation is an important consideration. Biological sample preservation normally emphasizes:

- a. Sample holding container
- b. Type of preservation used
- c. Sampling labelling information
- d. Holding time between sampling and analysis

The containers for biological sample material can be divided into two principal categories: glass and plastic. Both kinds of containers require

the use of proper chemicals to preserve the field biological materials. The chemical preservatives most often used by BMI personnel for general field preservation include formaldehyde and ethyl alcohol.

Field technicians make a practice of duplicating full data on a second label and packing it within the sample container so that at least one set of sample information is preserved. This labelled sample should go to BMI's project manager or laboratory supervisor with a completed Field Project Sheet.

7.2 Field Data Analysis

Field data sheets are filled out at each sampling station. The field measurements, along with the biological data collected, provide the data base for most field studies. Many analyses are performed in the laboratory. These include organism identification and length-weight measurements of organisms for productivity estimates.

A variety of techniques are used to insure the accurate count of organisms in a given sample. Macroinvertebrate samples, for example, are stained to increase the efficiency of the sorting process. Sieves (at least as fine-meshed as the sampling net) are employed to concentrate animals for later identification. Sample labels are kept with the sample until identification is completed to insure accurate sample handling.

Several taxonomic references are available and utilized by BMI personnel in order to insure positive identification of organisms. In addition, outside experts are regularly consulted in order to check identifications.

7.3 Functional Tests

BMI performs several types of field tests. These include: stream surveys, secondary productivity analyses, and bioaccumulation determinations. The accurate performance of these tests depends on the factors outlined above and the qualifications and experience of the sampling team. The latter is insured through a rigorous training program of all involved personnel and frequent checking of their performance by knowledgeable principals in the firm.

8.0 LABORATORY TOXICITY TESTING

8.1 General

8.1.1 Organism Handling

The accuracy and quality of laboratory tests depends on the health and well-being of the appropriate test organisms. The rigorous protocols for organism culture and maintenance (Section 6.0) are designed to insure the well-being and health of test organisms.

Organisms used in tests are handled with great care to insure a minimum amount of stress prior to testing. Any organism exhibiting signs of stress is not used for test procedures, nor are any which may be injured in the transfer process.

8.1.2 Reference Toxicants and Control Charts

The health of test organisms is checked routinely using reference toxicant studies. Each batch of test organisms obtained from an outside source is evaluated by reference toxicant studies. Organisms from in-house cultures are evaluated at least twice a month. If a reference toxicant study is failed, determined by control chart statistics, and investigation of the cause of failure is initiated. In addition, the test is repeated. All tests performed with organisms from the same batch as the failed reference toxicant are repeated. No testing for clients is performed until the problem is corrected.

A control chart is prepared for each reference toxicant study, and successive toxicity values are plotted to determine if the results are within prescribed limits. A running plot is maintained for the toxicity values from successive studies with a given reference toxicant.

8.1.3 Randomization Procedure

Randomization is a necessary aspect for insuring accurate data generation and interpretation in experiments. Therefore, care is given to insure the randomization of possible conflicting and extraneous effects.

In general, all tests utilize a sequential assignment of organisms to test containers in order to minimize effects due to different sizes, or quality of organisms among test containers. Also, test containers are randomized with respect to location in a given test and with respect to which test condition it is assigned. The protocols insure that the test is not biased with respect to differences among containers or their location with respect to each other.

8.1.4 Quality Control

A QCO (Quality Control Officer) supervises procedures affecting each aspect of laboratory toxicity testing such as effluent sampling and handling, condition of test organisms, condition of equipment, test conditions, instrument calibration, use of reference toxicants, record keeping and data evaluation. Data is recorded on log sheets or other designated forms and reviewed systematically from the beginning to the end of the testing procedure. Permanent records of data and relevant information are kept on file. Any questionable information at any time is reported to the QAO who initiates proper procedure for correction.

8.2 Toxicity Testing Procedures and Guidelines

BMI's acute toxicity testing methods are designed to conform to Peltier and Weber, 1985, as stated in EPA publication EPA/600/4-85/013, Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms, (3rd ed.). A step by step method is included in BMI's SOP Document (G.1) which is used for training of technical staff and is accepted as the uniform method of carrying out the definitive test procedure. Specific capabilities include methods for testing using freshwater and marine or estuarine invertebrates and/or fish. The individual SOP's for each species can be found in Section G.0 of the SOP Document.

Chronic test capabilities include algae and plant studies as well as invertebrate survival and reproduction tests and fish (marine and freshwater) survival and growth tests. Section H.0 of the SOP Document gives details on methods utilized by BMI staff for chronic toxicity testing using various species of organism. These methods conform to EPA/600/4-87/028 Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms as well as guidelines set for TSCA testing (EPA-1982).

9.0 DATA HANDLING AND ANALYSES

9.1 General

A most important aspect of quality assurance and quality control is precise reliable data. Information recorded concerning laboratory environment, culturing, and test procedures all have an impact on the final results of a project. To assure uniform data recording, a log sheet method of data collection is implemented at BMI whereby a specific log sheet is designed to facilitate the recording of specific information for analysis. Technical staff members keep concise records via log sheets of data concerning all aspects of laboratory functions. All entries on log sheets are initiated. The QCO checks that data is logged in properly and on time. Log sheets are compiled at the end of a project and pertinent data is reviewed and reported as the situation requires. The QAO is ultimately responsible for evaluating and reviewing all data and reports resulting from projects prior to reporting to clients or transmitting to permanent storage.

Laboratory audits are performed by the Laboratory Manager on a quarterly basis or more often if necessary. The BMI Quarterly Quality Assurance Audit form is filled out by the Laboratory Manager or a superior and is designed to assure that data recording, handling, analysis, and storage are being conducted properly. Data reports must also have the final approval of the Laboratory Manager.

9.2 Data Recording, Analysis, and Summarization

All internal laboratory data sheets have a heading in which the date, identifier, type, mode, duration, and species used for the particular test are indicated. This procedure reduces the frequency of misplaced data sheets. All data log sheets for a particular test are kept together on a clip board adjacent to the test containers to avoid misplaced data sheets. Each test container is labeled with the test identifier as well, to assure that the data for test containers are recorded on the proper data sheets. Once a given test is completed, the data sheets are photocopied and the original sheets are placed in a bound notebook with the appropriate identifier file marker. These notebooks are kept on permanent file in the data records area. The original chain of custody form for the water sample used in a test is kept with test results in the same notebook.

Data are analysed in order to obtain summary test results. The form of analysis depends on the type of test performed. The analysis procedures are available on IBM computer programs kept on file for each type of test. All computer-analyzed output (such as those used to generate LC50 values) includes a listing of the input values and test identifier in order to check the data input with the original data. This check is made by the Quality Control

Officer in charge of the test. Data which is analyzed manually is checked by the Quality Control Officer prior to data summarization.

Data for a given test are summarized using standardized summary sheets suited for the particular type of test. When statistical means are presented for biological data, they are given to one significant decimal place with accompanying estimates of the variability in the data. The variability may be expressed as 95% confidence limits, standard error terms, or coefficient of variation depending on the requirements of the test analysis performed.

Summary sheets are checked for accuracy by the Quality Control Officer prior to copying and storage. Correct summary sheets are photocopied and the original is placed with the original data log sheets and chain of custody sheet(s) in permanent data notebooks. Toxicity testing data is stored separately from field data.

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11.0 KEY PERSONNEL

NAME: Jerome M. Diamond

POSITION: Chief Scientist, Biological Monitoring, Inc.

SUMMARY OF QUALIFICATIONS:

Jerome Diamond has been a Technical Director of Biological Monitoring, Inc., since 1984. Dr. Diamond serves on several subcommittees under the ASTM E-47 committee on Aquatic Toxicology and Hazard Assessment and routinely reviews articles and grant proposals for international technical journals and federal agencies. He is a member of the Sigma Xi Honor Society, and has written numerous articles and reports in the field of aquatic ecology and toxicology.

EDUCATION AND TRAINING:

- Ph.D., Ecology and Stream Biology, 1984, University of North Carolina, Chapel Hill, NC
- M.S., Stream Ecology, Freshwater Biology, 1976, Oregon State University, Corvallis, OR
- B.A., Biology, 1973, Case Western Reserve University, Cleveland, OH

EMPLOYMENT HISTORY:

1984-Present	<u>Organization:</u> Biological Monitoring, Inc. <u>Position:</u> Chief Scientist <u>Responsibilities:</u> Direct all forms of aquatic biological testing for industries, municipalities, and regulatory agencies; Quality Control Officer; Data analyses and interpretation; technical report writing and recommendations; client interfacing. Oversee organism culture and lab maintenance.
1980-May 1984	<u>Organization:</u> University of North Carolina, Chapel Hill, NC, Biology Department <u>Position:</u> Research Assistant <u>Responsibilities:</u> Perform and report aquatic field research in Piedmont streams impacted by housing developments and agricultural runoff. Data collection analyses, and interpretation. Technical writing and literature search.
1979-1980	<u>Organization:</u> U.S. Forest Service, Mapleton, Oregon <u>Position:</u> Fisheries Biologist <u>Responsibilities:</u> Watershed research, assess impact of logging practices on water quality and fisheries habitat. Computer simulation, statistical analyses, field data collection. Interpretation of maps, report writing and modeling of instream impacts.
1977-1979	<u>Organization:</u> Oregon Department of Fish and Wildlife, Marine Section, Corvallis, Oregon <u>Position:</u> Fisheries Biologist <u>Responsibilities:</u> Fish physiologist research, ATPase and thyroxin analysis. Experimental design and data collection. Statistical analyses, report writing. Hatchery evaluations, literature search and Information Report.

NAME: Marolyn J. Parson

POSITION: Research Scientist, Biological Monitoring, Inc.

SUMMARY OF QUALIFICATIONS:

Marolyn Parson has been the Senior Research Scientist of Biological Monitoring, Inc. since 1988. Dr. Parson has been involved with environmental assessment and monitoring since 1984. She has experience both in utilizing and supervising approved EPA and Standard Methods to measure chemical, physical, and biological parameters commonly used to monitor water quality. She has written numerous articles and reports in the field of aquatic ecology, phycology, and toxicology.

EDUCATION AND TRAINING:

- Ph.D., Biology, 1988, Virginia Polytechnic Institute and State University, Blacksburg, VA
- M.A., Teaching in the Community College (Biology), 1973, Western Michigan University, Kalamazoo, MI
- B.A., Education (Biology major), 1964, University of Northern Iowa, Cedar Falls, Iowa

EMPLOYMENT HISTORY:

Nov, 1988-Pres	<u>Organization:</u> Biological Monitoring, Inc. <u>Position:</u> Senior Scientist <u>Responsibilities:</u> Writes grant proposals to government agencies such as EPA, NSF, and NIH. Prepares reports for clients for whom field studies, bioassays, etc., have been performed. Develops experimental designs for research projects involving one of our products, an automated biomonitor. Develops experimental plans for field studies. Develops toxicity reduction evaluation plans, environmental assessment statements, etc.
June, 1988-Sept, 1988	<u>Organization:</u> Olver, Inc., Blacksburg, VA <u>Position:</u> Laboratory Director <u>Responsibilities:</u> Supervised all laboratory activities. Hired and evaluated all laboratory personnel (lab technicians, field samplers, clerks, etc.) Wrote reports for clients. Advised clients about environmental problems. Set priorities for laboratory activities. Interpreted results for clients.
June, 1987-May, 1988	<u>Organization:</u> Virginia Polytechnic Institute and State University, Biology Department <u>Position:</u> Graduate Research Assistant <u>Responsibilities:</u> Planned and conducted monthly monitoring of Mountain Lake, Virginia. Assisted in lake management decisions. Assisted in establishing bimonthly biomonitoring of streams and wells in watershed where sewage treatment effluent is sprayed on forest vegetation. Utilized atomic absorption spectrophotometry to detect concentrations of selected metals. Utilized radioisotopes to measure photosynthetic rates and ammonia uptake rates of phytoplankton. Analyzed data using SAS and SPSS statistical programs. Utilized Utermohl settling chambers and wild inverted microscopy in algal identification and enumeration. Participated in aerial survey of watershed area using infrared photography. Utilized PC graphics packages to produce slides and transparencies for presentation of results. Utilized gas

chromatography for nitrogen fixation measurements.

Fall 1984-Spr 1987	<u>Organization:</u> Virginia Polytechnic Institute and State University, Blacksburg, VA <u>Position:</u> Graduate Teaching Assistant <u>Responsibilities:</u> Taught general Biology Laboratory, Principles of Biology Laboratory, and Phycology classes
1975-1984	<u>Organization:</u> Montgomery County Schools, Christiansburg VA <u>Position:</u> Biology teacher/Department Head <u>Responsibilities:</u> Taught biology classes and served as Science Department Head at Blacksburg High School.
1966-1969	<u>Organization:</u> Fort Dodge Community Schools, Fort Dodge, Iowa <u>Position:</u> Science Teacher <u>Responsibilities:</u> Taught life science classes at North Junior High, Fort Dodge, Iowa
1965-1966	<u>Organization:</u> Newton Community Schools, Newton, Iowa <u>Position:</u> Science Teacher <u>Responsibilities:</u> Taught life science classes at Central Junior High, Newton, Iowa.
1964-1965	<u>Organization:</u> Waterloo Public Schools, Waterloo, Iowa <u>Position:</u> Science Teacher <u>Responsibilities:</u> Taught general science at West Junior High School, Waterloo, Iowa

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NAME: Elizabeth B. Layman

EDUCATION: B.S., Biology, Virginia Tech, May 1988.

TRAINING AND PROFESSIONAL EXPERIENCE:

June 1988 to December 1989-Aquatic Biologist, Biological Monitoring, Inc. Duties and training include freshwater and marine indicator organism culturing, conducting acute effluent toxicity test with various fish and invertebrate species, performing quality assurance reference toxicant tests, routine physiochemical monitoring and laboratory maintenance.

December 1989-present-Lab Supervisor, Biological Monitoring, Inc. Responsible for overall operation of toxicity testing, laboratory, laboratory personnel, test scheduling for clients, and data analysis and reports.

NAME: Donald G. Mackler

EDUCATION: B.S., Biology, Minor, Sociology, Virginia Tech, 1983.

TRAINING AND PROFESSIONAL EXPERIENCE:

1988-present-Aquatic Biologist, Biological Monitoring, Inc. Duties and training include freshwater and marine indicator organism culturing, conducting acute effluent toxicity tests with various fish and invertebrate species, performing quality assurance reference toxicant tests, routine physiochemical monitoring and laboratory maintenance. Responsible for performing and coordinating short-term chronic Ceriodaphnia survival and reproduction test and fathead minnow larval survival and growth tests. Assists with aquatic field studies. Performs on-site flow-through toxicity studies and toxicity test data analysis.

APPENDIX E
Effective Porosity and Permeability Testing
The Earth Technology Corporation

AR302283

EARTH TECHNOLOGY
GEOMECHANICS LABORATORIES
QUALITY ASSURANCE PROGRAM
REVISION 0

DECEMBER, 1989

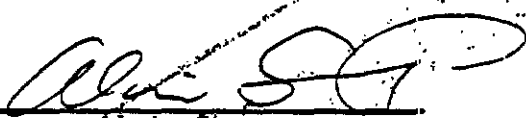
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EARTH TECHNOLOGY
GEOMECHANICS LABORATORIES
QUALITY ASSURANCE MANUAL

December, 1989

Approved: 

Manager, Geomechanics Laboratories

Approved: 

Alain Sharp
Corporate QA Manager

Issued To: _____

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AR302285

GEOMECHANICS LABORATORIES
QUALITY ASSURANCE MANUAL

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1.0 INTRODUCTION

Earth Technology Geomechanics Laboratories is a part of the Western subsidiary of The Earth Technology Corporation. The laboratories provide a broad range of testing services in a variety of sample matrices such as soil, sediment, and rock. The objective of the laboratory's QA/QC Program is to ensure that all data collected and processed through the laboratory are scientifically valid and properly documented.

The format and content of the Quality Assurance manual generally follows the recommendations made in "Guidelines and Specifications for Preparing Quality Assurance Program Plans" (QAMS-004/80) and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (QAMS-005/80). The program meets the requirements of ANSI/ASME NQA-1 as applicable to geomechanical testing. It is a generic plan designed to provide a description of the procedures that will be used to document and report the data generated.

Laboratory (Test) procedures have been developed based on ASTM and Corps of Engineer procedures and EPA methods. These procedures outline any modifications of the original procedure or method, and also document any in-house generated procedures. No deviations from the SOPs or the QA manual are permitted except those specified under contractual agreements.

The Quality Assurance Program defines the authority and responsibilities of laboratory personnel, and provides the requirements by which the quality of tests and services will be achieved and verified. The QA department is organizationally and functionally independent from the laboratories, with the laboratory QA coordinator reporting directly to the corporate QA manager. The laboratory QA coordinator will have the direct responsibility for the development, verification, and continued operation of the QA program in cooperation with the manager and staff of the laboratory.

2.0 ORGANIZATION

The Geomechanics Laboratories are organized into two major laboratories: Soil Mechanics Laboratory (SML) and Environmental Geomechanics Laboratory (EGL). Testing of noncontaminated soil, sediment and rock is performed in the SML. Contaminated materials are tested in the EGL. See Figure 2-1 for the laboratory organization chart.

2.1 Quality Related Responsibilities

Quality related responsibilities within the laboratory are as follows:

Laboratory Director

- Reports directly to the President of The Earth Technology Corporation (Western)
- Provides overview of activities in all the laboratories.

Laboratory Quality Assurance Coordinator

- Reports directly to the Earth Technology Corporate QA Manager
- Is responsible for verifying and assisting in the QA program in the laboratory
- Trains personnel in Quality Assurance practices
- Verifies implementation of a calibration and preventive maintenance program for test equipment
- Participates in inter-laboratory accreditation and proficiency programs
- Performs periodic audits of the laboratory
- Provides annual QA reports to management.

Laboratory Health and Safety Officer

- Reports directly to the Corporate Health and Safety Officer
- Assesses potential hazards associated with specific testing programs and EGL activities in general

- Provides operating guidelines and procedures to mitigate any potential hazards
- Provides health and safety training for all individuals participating in EGL testing operations
- Monitors and enforces health and safety procedures and emergency response procedures
- Maintains health and safety records.

Laboratory Manager

- Reports to Laboratory Director
- Manages the laboratory's daily operations
- Approves changes in the Quality Assurance program and laboratory (test) procedures
- Provides technical overview of laboratory activities
- Responsible for implementing the laboratory QA program
- Checks and signs laboratory reports
- Evaluates and implements new testing techniques, procedures, and instrumentation
- Evaluates qualifications of personnel and approves certification on individual testing procedures.

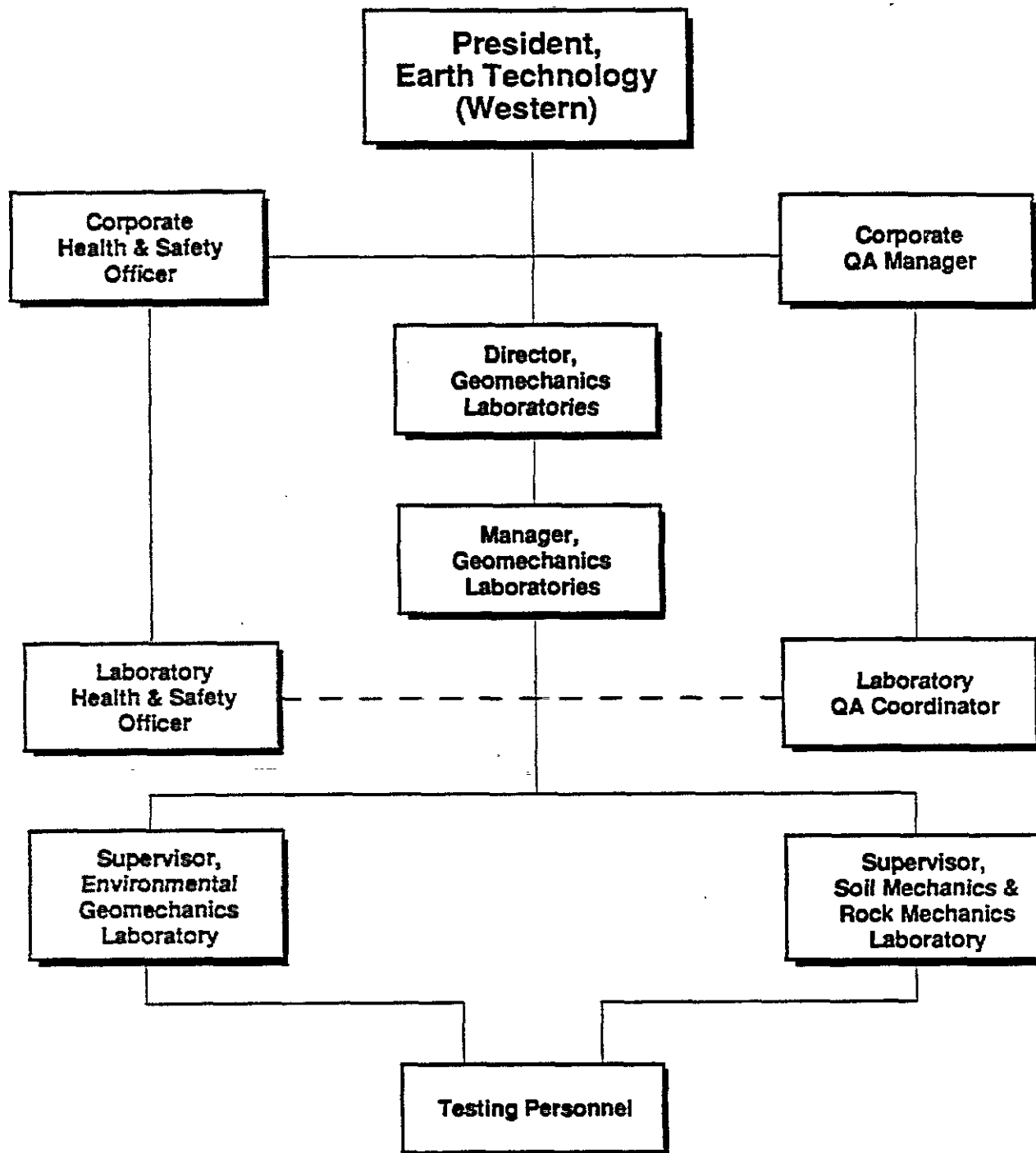
Laboratory Supervisors

- Report to the Laboratory Manager
- Supervise and document the training of personnel in laboratory operations and testing procedures
- Responsible for the log-in of samples received in their laboratory, chain-of-custody procedures and maintenance of sample inventory
- Organize and schedule the testing program

- Implement data verification procedures
- Supervise calibration program
- Supervise the preventive maintenance program
- Report all non-conforming situations to the Laboratory Manager and the Laboratory QA Coordinator.

Testing Personnel

- Perform the testing procedures and data recording in accordance with approved procedures
- Perform and document the calibration and preventive maintenance of test equipment
- Perform data processing and validation
- Immediately report instrument malfunction, calibration problems or other non-conformance to the Laboratory Supervisor.



Geomechanics Laboratories - Organization Chart
Figure 2-1

3.0 LABORATORY OPERATION

3.1 Facilities and Equipment

Earth Technology Geomechanics Laboratories is located at 18411 Gothard Street, Huntington Beach, CA, 92649. The laboratory facilities cover approximately 10,000 square feet of floor space, and are organized into sample receiving and storage, soils laboratory (clean lab), environmental laboratory for testing of contaminated materials with level D, level C and level B sections, and rock mechanics laboratory. A floor plan is presented in Fig. 3-1.

The Geomechanics Laboratories are equipped to perform conventional and specialized geotechnical and geochemical testing of clean and contaminated materials and waste solidification and bench processing of contaminated materials. The facility houses testing equipment, environmental monitoring and sample screening instrumentation, and support equipment (such as vacuum pumps and air compressors). The facility's geotechnical (physical) testing capabilities include index properties (grain-size distribution, Atterberg limits, etc.) compaction (standard and modified Proctor), triaxial hydraulic conductivity (permeability), shear strength evaluation (triaxial, direct, simple shear, static and dynamic), consolidation, compatibility testing, column and batch leaching, bench processing, waste solidification, stabilization, and fixation, among others. In addition, geochemical facilities are available to aid in sample, leachate and mix characterization.

3.2 Personnel Training

Personnel shall have known and documented training and/or work experience and, if required, minimum qualifications of education. The Laboratory Manager shall be responsible for initial evaluation of capabilities and qualifications of assigned personnel, and be responsible for periodic evaluation of assigned personnel. Documentation of qualifications and evaluations shall be retained in the laboratory files.

The training begins with an orientation process in which the new hire is informed of the corporate and laboratory operational practices. Laboratory training of new hires will include on-the-job training with evaluation. The corporation provides in-house technical seminars, and financial assistance is provided for elective studies, graduate, and undergraduate programs for interested employees.

Supervisory personnel shall train personnel under their jurisdiction as required to maintain suitable technical proficiency and expertise. Certification on a laboratory procedure requires that personnel be physically capable of performing the procedure, be familiar with technical aspects of the equipment and procedures, and be able to verify that the equipment is in proper working

condition and has been calibrated prior to use. Personnel shall also be tested by performing the procedure, recording the data, and performing required calculations as specified in the procedure. The testing shall be witnessed and the test results checked by the Laboratory Manager or his/her certified delegate, and acceptable performance shall be documented on a Capability Demonstration Form (Form 1B-1 or equivalent, Figure 3-2).

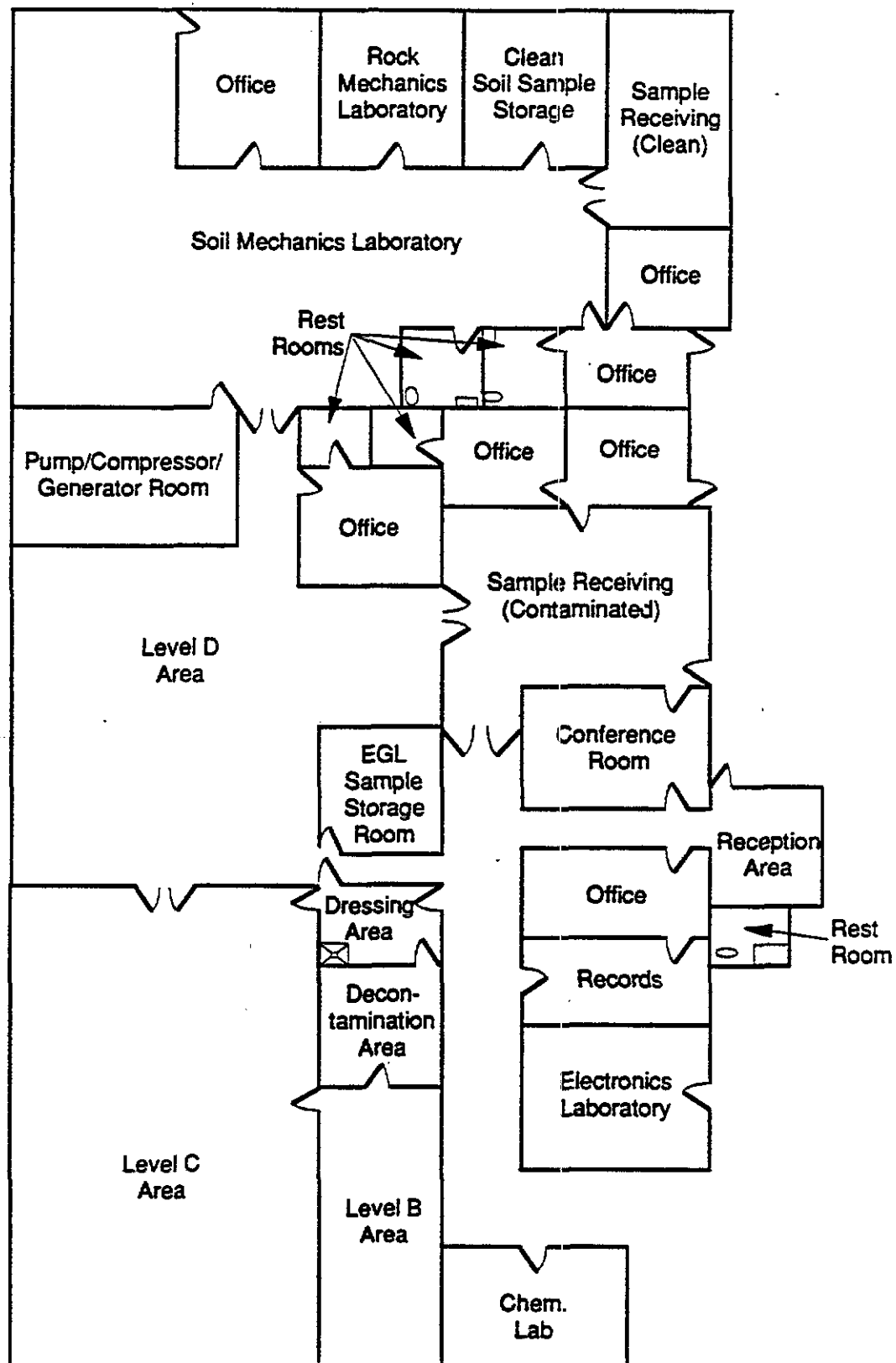
The laboratory manager shall evaluate an individual's education, experience, performance and/or training to verify that the individual meets the minimum requirements and is capable of performing the task per the appropriate procedure.

- o Certification shall be documented on the Qualification Evaluation Form (Form 1B-2 or equivalent, Figure 3-3) and include name of individual, procedure number(s) the individual is being certified to perform, education, experience, and training, results of capability demonstration, date of certification and date of expiration (2 years after date of certification), signature of laboratory manager and QA manager.
- o Recertification may be based on continued satisfactory performance of the activity in which case only Form 1B-2 need be completed. If the employee has not performed the activity within the past year, demonstration of capability is required, and both Forms 1B-1 and 1B-2 must be completed.

For certain projects outside certification, e.g., NICET (National Institute for Certification in Engineering Technologies), may be required.

Management at all levels has prime responsibility for the safety of all employees working under its supervision, and will conduct operations in a safe manner at all times.

All laboratory work shall be performed in accordance with the Earth Technology Corporation Health and Safety Manual. In addition, all work in the EGL shall be performed in compliance with the General Health and Safety Plan for Mechanical Testing of Contaminated Materials (TETC, December 1986). All personnel working in the Environmental Geomechanics Laboratory (EGL) must undergo a 40 hr Health and Safety training course.



Geomechanics Laboratories - Floor Plan
Figure 3-1

**Field and Laboratory Procedures
Capability Demonstration**

Name _____	
Position _____	
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____
Procedure No. _____	Procedure name _____
Pass ☐ Fail ☐	Evaluated by ☐ _____ Date _____

FORM 15-1
10/10/98

Field and Laboratory Qualification Evaluation

_____ has adequately demonstrated proficiency in the performance requirements and has satisfied the physical, education, and experience requirements for certification in conformance with procedure 1B for the following procedure(s):

No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____
No. _____	No. _____

Education, experience, and training: _____

Capability demonstration: _____

Date of certification _____ Signature _____
 Date of expiration _____ Signature _____
 Program QA/QC Officer

FORM 1B-2
5/1/88

4.0 PROCUREMENT AND SUBCONTRACTING

Before items or services can be acquired, a purchase order, subcontract or consultant agreement must be in place. The procurement process starts with a purchase requisition (PR).

Personnel preparing PRs shall conform with the requirements of the Corporate Procurement Procedures manual. If the requisition for items or services, e.g., calibration of equipment, includes QA/QC specifications, personnel preparing the PR shall include appropriate technical and QA requirements. The PR shall be submitted to the Laboratory QA Coordinator for review prior to issuance. QA review and approval must be documented by signature and date. PRs with QA requirements should include the following:

- o Statement of the scope of work to be performed.
- o Technical requirements including, as applicable, reference to specific regulations, procedures, or instructions, that describe the services to be performed or item to be procured. Technical requirements should be prepared by the appropriate technical personnel.
- o Quality Assurance Program requirements, requirements for special controls such as calibration, sample handling, etc.
- o Right of access to the supplier's facilities and records for inspection/surveillance or audit.
- o Documentation requirements, including the identification of documents to be supplied for information, review, or acceptance by Earth Technology and the time of submittal to Earth Technology.
- o Requirements for reporting nonconformance to Earth Technology.

If changes are made to the PR statement of work or QA requirements prior to purchase order or contract award, the final procurement documents for services or items shall be reviewed and approved by the appropriate technical and QA personnel.

Proposals shall be reviewed by technical personnel and for those with QA requirements, the proposals shall also be reviewed by QA personnel. The review shall include an evaluation of the capability of the proposers to meet the specified technical and QA requirements as appropriate.

Purchased services and items shall be evaluated for compliance with technical and QA requirements per Corporate Procurement Procedures. The receiving report

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shall be approved by technical personnel and by the Laboratory QA Coordinator if QA requirements were specified with PR.

5.0 SAMPLE CONTROL

This section outlines the handling of samples from receipt at the laboratory to disposal.

5.1 Chain of Custody

The purpose of the chain of custody procedures is to establish detailed written and legal documentation of all transactions in which the samples are transferred from the custody of one individual to another. When used, chain of custody forms and procedures are to be utilized from the time of sample collection until receipt into the laboratory. Every change of possession of the sample(s) must be documented on the chain of custody form. The chain of custody forms must also be used if the laboratory relinquishes control of the samples to sub-contractor labs or to return the samples to the originator.

Whenever samples are received into the laboratory, a sample tracking document must be completed. All samples received in the EGL shall be accompanied by a chain of custody form (see Figure 5-1 for an example). For samples received in the SML a chain of custody or sample inventory form (Figure 5-2) can be used. If no document is included with the samples, one will be filled out upon receipt. Client chain of custody documents can be used provided that proper identification of samples, date and time of collection and exchange, and identification of sender company is on the form.

5.2 Sample Receipt

If the samples are potentially contaminated the Sample Manager or his designee shall refer to the Health and Safety Plan and project specific addendum for special handling and storage instructions.

Upon arrival of the sample(s) at the laboratory, the sample shipping containers shall be inspected by the Sample Manager or his designee for damage that may have occurred during shipment. If several bagged or otherwise contained samples are in the shipping container they shall be inspected individually for damage during shipment. (Sample should not be removed from individual containers such as bags, rings, or tubes.) Any noticeable damage shall be recorded on the chain-of-custody or inventory form received with the sample.

Number of samples and sample numbers shall be verified against any inventory or chain-of-custody forms received with the samples. Receipt acknowledgement will be completed per received inventory or chain-of-custody requirements. Sender shall be notified of any discrepancies or damages.

assigned number. The label shall be securely attached to samples at all times or the sample may be placed in containers with the appropriate information. TETC assigned sample numbers shall consist of the final 6 digits of the project number, followed by a letter indicating the type of sample and consecutive numbers as shown in the following example. Sample types shall be abbreviated as follows:

- o Ring samples - R
- o Bag samples - b
- o Tube samples - T
- o Large bulk samples (drums or cans) - B

For example, for project number 88-205-2709 2 ring samples and 2 bag samples were received. Sample numbers are as follows: 052709R-1, 052709R-2, 052709b-1, 052709b-2.

For projects with NQA-1 or equivalent QA requirements, the samples shall be inventoried on the Inventory Control Sheet (Form L-4901), (Figure 5-3), and the storage location noted on the inventory control sheet.

Copies of the inventory control sheet and/or chain-of-custody or other receiving documents shall be filed as follows:

- o One copy to the project file
- o One copy to remain in the sample storage room
- o One copy for the Laboratory Supervisor's file

5.3 Sample Storage and Control

The storage area shall be secured and samples shall be placed in the storage room in a manner such that they can be easily retrieved. If samples require special storage conditions, written instructions are to be provided.

The Laboratory Supervisor shall implement a retrieval system whereby the flow of samples is controlled between storage, sample preparation/testing, and return to storage, if required. For projects with NQA-1 or equivalent QA requirements, specimens or portions of samples used in destructive testing shall be noted in the Inventory Control Sheet comments. Unused portions of samples shall be returned to storage until completion of the testing program. All sample retrieval/return shall be noted on the laboratory inventory control sheet and every process dated and initialed by authorized personnel. For standard projects use of samples may be tracked by Request For Static Lab Testing (Form LH-01). Sample(s) shall be stored until the Project Manager or his designated representative directs the disposal of the sample, or return to the client.

5.4 Test Request

The Project Manager shall designate the Laboratory (Test) Procedure to be performed on each sample and any necessary additional information by completing the Request for Static Lab Testing (Form LH-01) (Figure 5-4). The Laboratory Manager or Laboratory Supervisor is the Project Manager for testing program for external clients. The Test Request shall be approved by the Laboratory Manager or Laboratory Supervisor (this may be the same person that is requesting the tests), and forwarded to the Laboratory Supervisor for assignment of personnel to do the testing.

When all tests are completed the request form shall be placed in the laboratory project files with the test data. For projects with NQA-1 or equivalent requirements, the original test data and the request form shall be forwarded to the person requesting the test with copies placed in the laboratory files.

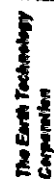
5.5 Sample Disposal

Non-contaminated (clean) samples shall be disposed of 60 days after testing. Contaminated samples shall be returned to the client after testing is completed with storage not to exceed 60 days. If the client requests extended storage of samples the duration shall be specified in writing by the Laboratory Manager and noted on the chain-of-custody form or Inventory Control Sheet.

All contaminated samples and expendables associated with testing contaminated samples shall be returned to the client or his designated disposal agent. Instructions for shipping of radioactive samples shall be designated in the project specific Health & Safety Plan Addendum. For samples that must be shipped by commercial carriers and can be packaged such that individual containers with samples weigh less than approximately 50 lbs., the procedures in laboratory procedure LP-490 shall be followed.

[illegible]

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GML QA Manual

Sample Inventory Form

AR302304

3

Figure 2-2

Project No. _____

Project Name _____

Sheet No. _____ of _____

Checked by _____ Date _____

[illegible]

• S = Shipped to client
• D = Disposed
• T = Destroyed during testing

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Corporation

1-4901
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GMIL QA Manual

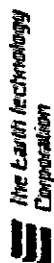
The Earth Technology Corporation

Inventory Control Form

AR302305

2

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(1) THE REQUESTOR SHOULD FILL OUT THE UNIT PRICES. FOR TESTS NOT CHARGED ON A UNIT PRICE BASIS, FILL OUT N/A. THE UNIT PRICES WILL BE CHECKED BY THE LAB SUPERVISOR

(5) IF COMPACTION TESTS ARE REQUESTED, THE DRY UNIT WEIGHT CAN BE EXPRESSED AS A PERCENTAGE OF MAXIMUM DENSITY AND THE MOISTURE CONTENT CAN BE EXPRESSED AS A PERCENTAGE ABOVE OR BELOW OPTIMUM MOISTURE.

The Earth Technology Corporation

The Earth Technology Corporation

6.0 Laboratory (Test) Procedures

The Laboratory Manager shall insure that approved procedures are employed by the staff. Table 6-1 provides a list of typical laboratory procedures. These procedures are based on EPA, ASTM and Corps of Engineers procedures. Current applicable procedures shall be made available to the staff and a copy maintained in the laboratory. These procedures have been reviewed and approved by the Manager, Geomechanics Laboratories and the Corporate QA Manager.

The following format shall be used when developing laboratory procedures:

- o A cover page listing the procedure name and number, the dates of each current page and approval by the Manager, Geomechanics Laboratories and the Corporate QA Manager.
- o The procedures shall include the following sections as appropriate:
 - 1. Introduction Brief statement of purpose, applicability, etc.
 - 2. Planning Include equipment and calibration requirements
 - 3. Methodology (Procedure) List required step by step instructions
 - 4. Records
 - 5. Safety List special safety precautions to be observed
 - 6. References
 - 7. Forms

Other sections can be added as the requirements of the procedure necessitates.

- o All pages shall list the procedure number and title, page number and total pages at the top of the page and date and revision number at the bottom of the page.
- o The procedures shall include reference to appropriate EPA guidelines, ASTM and Corps of Engineers Procedures, if used, including any deviations or options used, forms to be used to record data, and detailed methodology to be followed.

Revisions to laboratory procedures shall be reviewed and approved in the same way as the original procedure. Temporary project specific changes shall be specified in the project work plan.

In addition to these approved laboratory procedures, special procedures may be developed upon request to meet a client's specific project needs. Since these

procedures are project specific, they do not need to go through the regular review and approval procedure, but must be approved by the client before testing begins. Testing is also done in accordance with client provided special procedures when there is a contractual commitment to do so.

TABLE 6-1. LIST OF LABORATORY PROCEDURES

LP-110	Resonant Column & Cyclic Triaxial Tests
LP-115	B-Value Procedure
LP-120	Static Triaxial Testing
LP-122	Consolidated Undrained Static Triaxial Test (R or CU)
LP-123	Unconsolidated Undrained Static Triaxial Test (Q or UU)
LP-125	Unconfined Compressive Strength of Cohesive Soils (ASTM D 2166)
LP-126	Pocket Penetrometer Testing of Soils
LP-130	One-Dimensional Consolidation Properties of Soils (ASTM D 2435)
LP-131	One-Dimensional Swell or Settlement Potential of Cohesive Soils (ASTM D 4546)
LP-135	Expansion Test (consol. method)
LP-137	Expansion Index Test
LP-139	Shrinkage Factor of Soils (ASTM D 427)
LP-140	Moisture-Density Relations of Soils and Soil-Aggregate Mixtures using 10-lb Rammer and 18-in Drop (ASTM D 1557)
LP-141	Moisture-Density Relations of Soils and Soil-Aggregate Mixtures using 5.5-lb Rammer and 12-in Drop (ASTM D 698)
LP-145	Density of Soil in Place by Sand-Cone Method (ASTM D 1556)
LP-148	Density and Moisture Content of Soil in Place by Nuclear Method (ASTM D 2922 & D 3017)
LP-150	Specific Gravity of Soils (ASTM D 854)
LP-155	Specific Gravity and Absorption of Coarse Aggregate (ASTM C 127)
LP-160	Liquid Limit, Plastic Limit and Plasticity Index of Soils (Atterberg Limits) (ASTM D 4318)

TABLE 6-1. LIST OF LABORATORY PROCEDURES
(Continued)

LP-170	Relative Density (ASTM D 4253 and D 4254)
LP-175	Relative Density (graduated cylinder method)
LP-180	Laboratory Permeability Test (constant head) (ASTM D 2434)
LP-181	Permeability Test Under Back Pressure (Triaxial Permeability Test)
LP-182	Constant-Head Permeability Test
LP-183	Falling-Head Permeability Test with Permeameter Cylinder
LP-190	Particle Size Analysis of Soils (Sieve and/or Hydrometer) (ASTM D 421 and D 422)
LP-190AZ	Particle Size Analysis (Mechanical) Army Corps Manual EM 110-2-1906 App V
LP-191	Sieve or Screen Analysis of Fine and Coarse Aggregates (ASTM C 136)
LP-192	200 Wash Method (ASTM D 1140)
LP-193	Visual Classification of Soils (ASTM D 2488)
LP-194	Estimated Engineering Classification of Soils (ASTM D 2487 and D 2488)
LP-195	Sand Equivalent Test (ASTM D 2419)
LP-200	Moisture-Density Determination
LP-201	Moisture Content (ASTM D 2216)
LP-202	Capillary-Moisture Relationships for Fine-Textured Soils by Pressure-Membrane Apparatus (ASTM D 3152)
LP-203	Capillary-Moisture Relationships for Coarse- and Medium-Textured Soils by Porous-Plate Apparatus (ASTM D 2325)
LP-204	Determination of Organic Matter in Organic Soils (ASTM D2974)

TABLE 6-1. LIST OF LABORATORY PROCEDURES
(Continued)

LP-205	Determining Clearance Ratios of Pitcher Tubes
LP-206	Volumetric Determination of Cylindrical Samples by Circumference Measurement
LP-210	Moisture Determination Employing Speedy Moisture Device
LP-220	Direct Shear
LP-240	California Compaction Test
LP-250	Bearing Ratio of Laboratory Compacted Soils (California Bearing Ratio) (ASTM D 1883)
LP-300	Undisturbed Block Sampling
LP-350	Crumb Test (COE EM-1110-2-1906, App. XIII)
LP-360	Pinhole Erosion Test (COE EM-1110-2-1906, App. XIII)
LP-370	Dispersion Characteristics of Clay Soil by Double Hydrometer (Double Hydrometer) (ASTM D 4221)
LP-380	Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (Aggregate Soundness Test) (ASTM C 88)
LP-390	Los Angeles Abrasion Test
LP-400	Sample Remolding
LP-401	Harvard Miniature Compaction (ASTM D 4609)
LP-450	Determination of Percent Calcium Carbonate
LP-451	pH Test (ASTM D 1884)
LP-452	Soil pH Determination (EPA 9045)
LP-490	Sample Handling, Shipping and Storage in the Laboratory

TABLE 6-1. LIST OF LABORATORY PROCEDURES

(Continued)

LP-500	Qualifying and Certifying Inspection and Testing Personnel
LP-501	Qualifications and Certification of Personnel to Perform Laboratory Procedures
LP-510	Drained Triaxial Compression Test (Without Pore Pressure)
LP-515	Triaxial Compression Test Data Reduction
LP-520	Drained Triaxial Compression Test (With Pore Pressure)
LP-525	Permeability Measurement (Transient Pulse Method)
LP-530	Compressional Wave Velocity Measurement
LP-540	Electrical Resistivity Measurement (4-Terminal Method)
LP-550	Rock Moisture Content Determination
LP-560	Thermal Conductivity Measurement (Transient Method)
LP-570	Axial Compression Test Specimen Preparation and Measurement
LP-580	Rock Core Sample Storage and Test Request
LP-620	Petrographic Evaluation (Rock Salt Core)
LP-800	Control of Laboratory Chemicals

TABLE 6-2. REFERENCES

- American Society of Agronomy and Soil Science Society of America, Methods of Soil Analysis Part 1, Physical and Mineralogical Methods, Edited by Arnold Klute et. al., 1986.
- American Society of Agronomy and Soil Science Society of America, Methods of Soil Analysis Part 2, Chemical and Microbiological Properties, Edited by A.L. Page et al., 1986.
- Annual Book of ASTM Standards, latest revision Volumes 4.02, 4.03, 4.05, 4.08, 14.01, 14.02.
- Corps of Engineers, Army Corps Manual, Laboratory Soil Testing, Engineer Manual No. 1110-2-1906, Rev. 1980.
- U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW 846, 3rd edition, 1986.

7.0 SOFTWARE QUALITY ASSURANCE

Documentation and control of all software programs acquired from outside sources as well as programs developed internally shall be handled per Section 7.1 through 7.5 of this manual. These requirements do not apply to software packages used for purposes other than scientific analysis, data acquisition, test control or database management, thus excluding for instance all word processing programs, spreadsheets and language compilers.

For laboratory operations documentation, calibration, verification and use of data acquisition/test control programs are of utmost importance.

7.1 Documentation

Documentation including program description, user instructions, program listing and test runs shall be prepared using the appropriate forms, which can be obtained from the Computer Department Manager. Program listing is waived in the case of machine language programs, and shall be noted on the documentation status sheet.

The program documentation and verification for all computer programs shall be reviewed by personnel to verify that the program documentation is complete and accurate and the program, documentation, and verification meet the requirements. Approval shall be documented by completing, signing, and dating the Computer Program Review and Approval Form and signing and dating the review and approval line of the Status Sheet.

7.2 Calibration

In order to verify that reliable data are collected and that the program correctly executes the commands to the test equipment, it is important to calibrate that entire system in its final configuration, including the computer. The computer should first be calibrated separately. The calibration can be performed as outlined in Section 7.5. The methodology shall be described and only calibrated instruments traceable to NBS standards or natural physical constants shall be used as calibration sources.

Computer programs used to control a testing sequence need only be calibrated as part of the program development and verification. A data acquisition program shall be recalibrated every 12 months to verify that the system is still performing properly. Records of this calibration shall be filed with equipment calibration records.

7.3 Verification

Program verification shall consist of calibrating the system and running a test case. A test case shall be run and results shall be compared with experimental measurements (e.g., calibrated pressure gauges). Documentation shall include a printout showing the output from the test case, a description of the test case, and discussion of the results, their reliability, and how well the program functioned.

7.4 Control

All computer programs shall be logged in the computer program library file. Computer programs shall be identified by version number and revision number. Any time a change is made to a program, the program will receive a new revision number. For major changes, the program will be identified with a new version number. Programs obtained from outside sources shall follow this procedure as closely as condition permits.

If the code and/or documentation require modification or a program user encounters a problem with the program, they shall initiate a Computer Program Problem/Change Report. After the change has been made and verification performed, the library file is updated and users of the program notified of the changes.

7.5 Use of Acquisition/Test Control Programs

Documentation of all computer analyses and test runs should include a listing of input data or data source and/or input commands and results. To facilitate reconstruction of a sequence of computer runs, the documentation should include the reason for each run. In such cases, the output need only contain input data that was changed.

All computer output should be identified by:

- a) Program name
- b) Version and revision number
- c) Project number
- d) Time and date of run
- e) Place for signature of person running the program
- f) Place for signature of person checking the output.

Requirements a) through f) should be printed out by the program. If provisions for printing any part cannot be made, headings and lines should be printed and information filled in by the person running program. For full screen interactive programs, such as Lotus 1-2-3, provisions should be made in the file for printing out the information.

All other support documentation, such as code input data including databases and original sources/references of and assumptions used to obtain such data, hand calculations, etc., should be properly identified. All documents shall be filed in the project file.

The computer output shall be checked by qualified personnel. This check shall be made to ensure the following:

- The input data are in agreement with original data source or are from an approved data base as appropriate
- The computer program executed the program and the results are reasonable
- The results are properly identified per Section 4.10.1.

The person performing the check shall sign and date the results.

In addition, calibration checks shall be performed. Before starting data acquisition, the system shall be tested to verify that the output matches the input within tolerances. If data output is monitored on a separate calibrated instrument (e.g., x-y recorder) or if a zero channel for the A to D convert system is recorded, no further documentation is required. In other cases, a test shall be performed in which a known input is applied and compared to the output. If the result is not within tolerances (depending on calibration specifications for the instruments involved), corrective action must be taken. The results of the calibration check shall be properly identified and included in the documentation.

8.0 EQUIPMENT CALIBRATION AND PREVENTIVE MAINTENANCE

The calibration program at Earth Technology Geomechanics Laboratories is equipment specific and will differ according to prescribed methodologies. Calibration procedures, at a minimum, include the equipment to be calibrated, the reference standards used for calibration, the calibration techniques and the sequential actions, acceptable performance tolerances, frequency of calibration, and calibration documentation format.

All tools, gages, instruments, and other measuring and testing equipment used in the laboratory to measure test parameters and results shall be calibrated. Rulers, tape measurers, levels and other such devices need not be calibrated if normal commercial equipment provides adequate accuracy, but shall be maintained in good working condition.

8.1 Calibration/Service Specifications

Calibration/Service Specifications (C/SS) shall be prepared for all laboratory equipment requiring calibration. The Laboratory Manager shall assign personnel to be responsible for performing the calibrations or procure outside calibration where calibration cannot be performed in-house. The C/SS manual shall be available to all personnel performing calibrations, and a copy maintained in the laboratory. New C/SS shall be submitted for inclusion in the C/SS Manual upon acquiring equipment not covered by existing C/SS. The periods between calibrations (calibration interval) shall be determined based on equipment stability characteristics, required accuracy, intended use, current professional and industrial practices and manufacturer's recommendations. Practices for equipment maintenance and service should be noted on the C/SS and the maintenance schedule should be based on intended use and manufacturer's recommendations.

All C/SS shall be reviewed and approved by the Manager, Geomechanics Laboratories and the Corporate Quality Assurance Manager. Revision to a C/SS shall be initiated by Earth Technology personnel if any information in the C/SS including calibration procedure must be changed for any reason. Revisions must be reviewed and approved. C/SS Forms 12A-1 and 12A-2 (Figures 8-1 and 8-2) or equivalent (Form 12A-2 required only for equipment being calibrated in house) shall be completed for all equipment requiring calibration.

8.2 Inventory

Each laboratory performing testing shall maintain records of all equipment which require calibration. These records shall indicate the date of the last calibration, the item name, C/SS number, serial number or identification number, and the date the next calibration is due.

8.3 Calibration

Equipment requiring calibration shall be submitted for calibration before current calibration expires and/or calibrated daily or before use as specified in the appropriate C/SS. Equipment shall be recalibrated prior to use if mishandling has occurred or is suspected, or if the accuracy is suspected. If any equipment is consistently found to be out of calibration, it shall not be made available for use, and shall be repaired or replaced, as necessary.

In-house calibrations shall be performed by qualified personnel. The calibration shall be performed per the C/SS, using standards that are traceable to nationally recognized standards. If no nationally recognized standards exist, the basis for calibration shall be documented. The calibration shall be documented on Form 12A-3 or Form 12A-4 (Form 12A-4 is for recalibration requiring only a visual check) (Figures 8-3 and 8-4) or similar forms including the same information. If calibration is to be subcontracted, the procurement documents shall include requirements to comply with the appropriate C/SS.

8.4 Calibration Labels

All equipment (e.g., measuring devices, gages, test instrumentation, graphical recorders) shall bear a calibration label in plain view that shall indicate the following:

- o If no calibration or services are required, the device shall be marked "No Calibration Required." This is not required for rulers, tape measures, levels, and other such devices if normal commercial equipment provides adequate accuracy.
- o Equipment requiring daily calibration or calibration prior to use shall be marked "Calibrate daily per C/SS ____" or "Calibrate prior to use per C/SS ____", as appropriate, and the C/SS number to be used for calibration noted on the tag.
- o If periodic calibration is required, the calibration label shall indicate:
 - Equipment identification number

- Last calibration date
 - Next calibration due date
 - Initials of personnel performing the calibration.
- o When labeling is not acceptable due to the size or nature of an item, the calibration information shall be contained in the Calibration Record, which shall identify the serial number of the item, and/or be recorded on the equipment storage container.

Any instrument or measuring device noted with expired calibration shall be marked with red tag or tape and placed out of service until recalibrated.

8.5 Out of Calibration Equipment

If, during calibration, equipment is found to not meet calibration specifications, an analysis of the data obtained during the "out of calibration" status of the equipment shall be made and documented on Form 12A-5 (Figure 8-5) or equivalent and approved by the appropriate technical/laboratory manager and QA manager to determine the validity of, and ability to use the data.

8.6 Preventive Maintenance

Earth Technology Geomechanics Laboratories shall perform periodic preventive maintenance and service as appropriate for the type of test equipment in use. Schedule of maintenance shall be based on recommendations by the manufacturer and laboratory experience with the equipment. Simple operations are the responsibility of each technician, but more difficult and intricate maintenance operations are to be left to the instrument manufacturer's service representative or other qualified individuals.

No. _____
Dated _____

Calibration/Service Specification

Item name _____ Model number _____
 Manufacturer _____ Classification _____
 Calibration source _____ Maximum interval _____
 Maintenance _____ Maximum interval _____
 Prepared by _____ Approved by _____ Date _____
 Approved by _____ Date _____

Description		
Operational requirements		
	Calibration specification	
Characteristic/range	Tolerance	Condition/limitation
Maintenance specification		

FORM 12A-1
10/10/86

GML QA Manual

Calibration/Service Specification Form

1-80

Figure 8-1

Procedure :

C/SS No.

Dated

FORM 13 A-2
10/10/88

Calibration Data Record

Serial No. _____ Item name _____
 I.D. No. _____ Manufacturer _____
 C/SS No. _____ Model _____
 Calibration date _____ Next date due _____
 Calibrated by _____
 Standards used _____

Adjustment required Yes _____ No _____

Characteristic	Tolerance	Measured values	
		Before adjustment	After adjustment
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Limitations _____
 Condition comments _____
 Repair/rejection comments _____

FORM 12A-3
6/30/89

 The Earth Technology Corporation	GML QA Manual
Calibration Data Record	

2-90

Figure 8-3



Analysis of Data Obtained from Equipment Out of Calibration

Calibration information

Description of equipment _____

C/S No. _____ Equipment serial or I.D. No. _____

Description of characteristics found out of calibration spec. _____

Reported by _____ Date _____

Analysis

Data Obtained with equipment since last calibration _____

Effect on data _____

Corrective action required _____

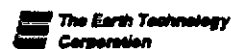
By _____ Date _____

Approved by _____ Date _____

Approved by _____ Date _____

Program QA/QC officer

FORM 12A-5
6/30/99



GML QA Manual

Analysis of Data Obtained from
Equipment Out-of-Calibration Form

2-80

Figure 8-5

9.0 DATA REDUCTION, VALIDATION, AND REPORTING

9.1 Data Reduction

The laboratory technicians are responsible for obtaining data and generating calculated results. Data reduction includes computer generated data as well as hand calculated data. All data shall be recorded on the appropriate test data sheets as indicated in the laboratory procedure. Documentation of the calculation process is required in order for the reviewer to verify the validity of the data reduction process.

9.2 Data Validation

At the bench level, data validation starts by cross-checking all sample identification numbers on sample bottles/tubes/rings/bags, test request sheets and instrument output. The technicians responsibilities further include ensuring that the instrument is calibrated and is responding in the appropriate manner before starting the test. The technicians will also exercise their own expertise in evaluating the data generated.

The second level review (check) will be performed by the laboratory supervisor or another qualified technician and includes verification of calculations and completion of test data sheets, including signature and date. The reviewer (checker) shall sign and date the test data sheet.

9.3 Data Reporting

A preliminary report is generated and after review by the supervisor, it is returned to data processing for preparation of a final report. The final report is signed by the laboratory manager who reviews the entire report for clarity and correctness of data.

All final data summaries are presented to the customer after the data reviews have taken place and signatures have been recorded.

The laboratory report will include, at a minimum, the following:

- The client project number (if given) and the laboratory job number
- Report date
- Test methods
- Signature of laboratory manager

Included in the laboratory report are Test Results Summary sheets. These contain at a minimum:

- Sample identification
- Sample depth (if known)
- Test Results

Figure 9-1 provides an example of a test results summary sheet.

(Method USEPA 9100)

PROJECT NAME: ABC Landfill Closure

TEIC NO.: XX-XXX-XXXX

CLIENT PROJECT NO.: XX-XXXX

CLIENT: XYZ ENVIRONMENTAL CORP.

REPORT DATE: MM-DD-YY

SUMMARIZED BY: **E.T. Employee**

LABORATORY MANAGER : E.Y. Lab Manager

[illegible]

THE EARTH TECHNOLOGY CORPORATION

GHM QA Manual

Example of Test Results Summary Sheet

2. 2.

Figure 9-1

10.0 MANAGEMENT OF RECORDS

Records are maintained in several places in the laboratory. Project files containing customer provided documentation and all laboratory generated chain-of-custody documentation, test requests, data sheets, and test reports are maintained in document files according to job site. These files are kept in numerical order based on laboratory assigned project number. Files are retained for a minimum of 5 years and then discarded. Laboratory reports will be stored indefinitely.

Documentation of personnel qualifications are kept on file in the laboratory. This shall include evaluation of qualifications, performance evaluations including test data sheets, any internally administered examinations, results of externally administered examinations, and final certification.

A sample log-in logbook is kept in the shipping and receiving area of the laboratory. This logbook keeps a running record of all jobs received into the lab.

Calibration data sheets will be filed in the calibration file whenever a calibration is performed. These data sheets will be retained for 5 years. The computer database shall be updated at least monthly. A monthly calibration due report shall also be generated. The same database serves as the equipment inventory.

11.0 NONCONFORMANCE AND CORRECTIVE ACTION

A nonconformance exists if there is a deviation from or noncompliance with contract and subcontract specifications, the QA program, approved procedures, work plans or other project requirements. Nonconformance also include major errors in documented analysis, data or results, and deficiencies in documentation. Regardless of the cause, any activity in the laboratory which adversely affects data quality is considered a nonconformance.

Personnel who identify a nonconformance should report the condition by completing Part A of the Nonconformance Report (NCR) (Form 15A-1, Figure 11-1) and distributing the NCR to the supervisor, and laboratory manager and laboratory QA coordinator. For projects that have an assigned project manager and program (project) QA/QC officer, the responsibilities of the laboratory manager and laboratory QA coordinator shall be handled by these individuals, respectively.

The supervisor, laboratory manager and laboratory QA coordinator shall evaluate the nonconformance and document this review on Part B of the NCR. The supervisor shall recommend corrective action, which shall be reviewed and approved by the laboratory manager and the laboratory QA coordinator, and documented on Part C of the NCR.

The approved corrective action shall be implemented by appropriate personnel, and verification and approval documented by the laboratory manager and laboratory QA coordinator on Part D of the NCR. Data generated shall be evaluated for impact of the nonconformance. If client notification is required, the laboratory manager shall submit a written report of the nonconformance to the client and obtain concurrence from the client of proposed corrective action. If the nonconformance has any impact on previously reported data, all individuals and organizations affected shall be notified.

Nonconformance Report (NCR)

Project _____ Project No. _____

Activity _____ Location _____

Part A

Description of non-conformance

Personnel reporting non-conformance _____ Date _____

Part B

Evaluation of non-conformance

Work stoppage required Yes _____ No _____ Significant condition adverse to quality Yes _____ No _____
Impacts previous data/reports Yes _____ No _____

Remarks _____

Evaluated by _____ Date _____ Title _____

Approved by _____ Date _____

Project Mgr.

Program QA/QC officer

Part C

Recommended corrective action/disposition _____

Recommended by _____ Date _____ Title _____

Approved _____ Date _____

Project Mgr.

Program QA/QC officer

Part D

Corrective action/disposition completed by _____ Date _____

Remarks _____

Corrective action approved and NCR closed by:

_____ Date _____

Project Mgr.

Program QA/QC officer

FORM 15A-1
7/1/88

12.0 QUALITY ASSURANCE AUDITS

In-house quality assurance audits are performed every six months by the Laboratory QA Coordinator or QA Manager. Audit reports are generated by the auditor with copies to be reviewed by the Laboratory Director/Laboratory Manager and supervisors. The purpose of the audit is to verify implementation of Earth Technology Geomechanics Laboratories QA program. Nonconformances that have occurred since the last audit and previous Quality Deficiencies Notices should be discussed with each supervisor and what corrections were made to remedy each situation. If necessary, quality assurance procedures can be changed to prevent some nonconformances from recurring.

The following should be checked during the audit:

- chain-of-custody procedures
- sample receiving and login procedures
- sample storage area
- procedures to prevent sample contamination
- storage and security procedures for laboratory and samples
- safety procedures
- conformance to written laboratory (test) procedures
- sample and data control systems
- procedures for handling and disposing of hazardous materials
- calibration status of equipment
- maintenance schedule and status of equipment
- procedures for data handling, reporting and filing
- training of new technicians
- corrective actions

If a deficiency is found during an audit, the auditor shall complete a Quality Deficiency Notice (QDN) (Figure 12-1, A and B), and the deficiency shall be discussed with responsible management in sufficient detail to ensure that the problem is clearly understood. These QDNs shall be included with the audit report. Responses to QDNs shall be provided by the Laboratory Manager within 30 days of audit report issuance. The auditor shall review the response and determine whether it is satisfactory. If the response and completion of corrective action taken are satisfactory, the auditor shall close the QDN and notify the laboratory manager of this action. If either the response or corrective action is unsatisfactory, the auditor shall request an additional response.

From time-to-time, external audits are performed by various clients or government agencies, (i.e., State of California, EPA, DOD, etc). Any deficiencies found during these audits shall be evaluated and addressed by the Manager, Geomechanics Laboratories in cooperation with the Laboratory QA Coordinator. The response to these audits shall be documented, and both the audit reports and responses to deficiencies shall be filed by the Laboratory QA Coordinator.

Quality Assurance requirements shall be specified in the procurement documents for outside laboratories and calibration subcontractors. The laboratory's or subcontractor's QA program shall be reviewed by Earth Technology QA personnel prior to start of work. An audit may be performed by Earth Technology to verify compliance with QA requirements, if appropriate. Upon completion of the audit, the auditor will discuss any nonconformance with responsible management. An audit report including Quality Deficiency Notices, if any, will be prepared and distributed to the subcontracting laboratory and the Manager, Geomechanics Laboratories. Responses to QDNs shall be reviewed and accepted by Earth Technology prior to start of work.

Quality Deficiency Notice

2. Project _____	1. QDN number _____
4. Activity _____	3. Project number _____
6. Controlling document _____	5. Location _____
7. Requirement _____	

8. Description of deficiency _____	

9. Reported by _____	10. Date _____
11. Discussed with _____	12. Date _____

Response:

13. This section to be completed by responsible organization and returned to Earth Technology QA by _____ (Date).

14. Corrective action (including action to prevent recurrence and root cause determination).

15. Scheduled completion date _____ 16. Signed _____ Date _____

FORM 16A-3
5/1/88

Page 1 of 2

 The Earth Technology Corporation	GML QA Manual
Quality Deficiency Notice (QDN) 1 of 2	
1-86	Figure 12-1a

Quality Deficiency Notice

Evaluation of response:

QDN number _____

17. This section to be completed by quality assurance department:

First response

☐ Satisfactory

☐ Unsatisfactory

Remarks _____
_____ Evaluated by _____ Date _____

Second response

☐ Satisfactory

☐ Unsatisfactory

Remarks _____
_____ Evaluated by _____ Date _____

Third response

☐ Satisfactory

☐ Unsatisfactory

Remarks _____
_____ Evaluated by _____ Date _____

18. Corrective action verified

☐ Yes

☐ N/A

Remarks _____

Verified by _____ Date _____

19. Quality deficiency notice closed on _____
By _____

FORM 18A-3
5/1/88

Page 2 of 2

 The Earth Technology Corporation	GML QA Manual
Quality Deficiency Notice (QDN) 2 of 2 3-80 Figure 12-1b	

13.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

A quality assurance report will be prepared by the Laboratory QA Coordinator and submitted annually to the Director, Geomechanics Laboratories, and the Corporate QA Manager addressing status of or changes to the QA Program Manual, significant QA problems, accomplishments and recommendations, results of audits, summary of QA training and measures of data quality.

14.0 MANUAL REVISION AND UPDATING

The Earth Technology Geomechanics Laboratories QA manual will be reviewed at least once every two years by the Corporate QA Manager to determine the need for revisions. Any laboratory personnel can suggest revisions to the manual such as changes, additions or deletions. Such suggestions should include the reason for the revision and a proposal of statements for the revised portion of the manual. The proposed revisions shall be reviewed by the Corporate QA Manager, Laboratory QA Coordinator, and Manager, Geomechanics Laboratories, and if approved, new pages will be prepared using the same page numbers. The replacement pages will be dated as of the revision. A new cover page shall be prepared and signed by the Manager, Geomechanics Laboratories and the Corporate QA Manager.

The Earth Technology Geomechanics Laboratories QA manual and revisions thereto shall be distributed as a controlled document per Section 15.0 of this document.

15.0 DOCUMENT CONTROL

The Earth Technology Geomechanics Laboratories QA manual, all laboratory (test) procedures, and the calibration/service specifications manual and any revisions thereto shall be distributed as controlled documents. The purpose of controlling these documents is to ensure that the current documents are available at the location where they are to be used, and that obsolete documents are withdrawn or identified as obsolete.

The Laboratory QA Coordinator shall issue the controlled documents as needed to Earth Technology personnel, clients and outside agencies. Each document shall be assigned a control number and this number and the recipient's name shall be logged by the Laboratory QA Coordinator. An acknowledgement of receipt shall be signed by the recipient and filed by the Laboratory QA Coordinator.

The recipient of the document shall return the document when the need for its use ends or upon cessation of employment, and the return shall be logged by the Laboratory QA Coordinator.

Uncontrolled copies of the documents may be issued if they are clearly marked "UNCONTROLLED". However, these documents cannot be used for performance of an activity, such as laboratory testing, or calibration.