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FINAL SAMPLE HANDLING AND ANALYSIS PLAN REMEDIAL INVESTIGATION VERIFICATION STEP MCRD PARRIS ISLAND SC

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FINAL
SAMPLE HANDLING AND
ANALYSIS PLAN
REMEDIAL INVESTIGATION
VERIFICATION STEP
MARINE CORPS RECRUIT DEPOT
PARRIS ISLAND, SOUTH CAROLINA

Report to

SOUTHERN DIVISION
NAVAL FACILITIES
ENGINEERING COMMAND
Charleston, South Carolina

McClelland

FINAL
SAMPLE HANDLING AND ANALYSIS PLAN
REMEDIAL INVESTIGATION
VERIFICATION STEP
MARINE CORPS RECRUIT DEPOT
PARRIS ISLAND, SOUTH CAROLINA

* * *

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* * *

SOUTHERN DIVISION NAVAL FACILITIES ENGINEERING COMMAND

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March 1988

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Quality Control in Environmental Testing and Hazardous Waste Characterization, General Engineering Laboratories, Charleston, South Carolina

SHA1.0 INTRODUCTION

The field investigation for the MCRD Parris Island sites is described in the Work Plan. This plan presents the procedures that will be used to obtain the field data. The number and types of samples which will be collected at each site, and the analyses to be performed on each sample, are summarized in Table SHA1-1.

Table SHA1-1
Summary of Samples and Analysis
MCRD Parris Island Remedial Investigation

Site/Location	Sample Type	Number of Samples	Analyses
Site 1 - Incinerator Landfill	groundwater	4	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Dissolved metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); Indicator Parameters (TOC, pH, specific conductance).
	sediment	3	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Total metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); EP Toxicity (As, Cd, Cr, Pb, Hg).
Site 2 - Borrow Pit Landfill	groundwater	3	Priority Pollutants (volatiles, acid extractables, pesticides, PCBs); Dissolved metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); Indicator Parameters (TOC, pH, specific conductance).
	sediment	1	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Total metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); EP Toxicity (As, Cd, Cr, Pb, Hg).

	surface water	1	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Dissolved metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); Indicator Parameters (TOC, pH, specific conductance).
Site 3 - Causeway Landfill	soil/sediment	8	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Total metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); EP Toxicity (As, Cd, Cr, Pb, Hg).
	surface water	8	Priority Pollutants (volatiles, acid extractables, base/neutral extractables, pesticides, PCBs); Dissolved metals (As, Ba, Be, Cd, Cr, Pb, Hg, Se, Ag); Indicator Parameters (TOC, pH, specific conductance).
Site 4 - Dredge Spoils Area Fire Training Pit	groundwater	3	Priority Pollutants (volatiles, acid extractables, base/neutral extractables); Indicator Parameters (TOC, pH, specific conductance); Dissolved metals (Cd, Cr, Pb).
	soil	5	Total metals (Cd, Cr, Pb); EP Toxicity (Cd, Cr, Pb)

Site 6 - Former Automotive Hobby Shop Spill Area	groundwater	1	Priority Pollutants (volatiles, base/neutral extractables); Indicator Parameters (TOC, pH, specific conductance); Dissolved metals (Cd, Cr, Pb).
	soil	2	Priority Pollutants (volatiles, base/neutral extractables); Oil and grease, dissolved metals (Cd, Cr, Pb).
Site 16 - Pesticide Rinsate Disposal Area	soil	6	Priority Pollutants (pesticides); Total metals (As, Cd, Cr, Pb).
Site 17 - Page Field AVGAS Tanks	groundwater	3	BTEX; Dissolved metals (Cd, Cr, Pb); Indicator Parameters (TOC, pH, specific conductance).
Site 18 - Page Field AVGAS Tanks	groundwater	3	BTEX; Dissolved metals (Cd, Cr, Pb); Indicator Parameters (TOC, pH, specific conductance).
Site 19 - MCX Service Station	groundwater	3	BTEX; Dissolved metals (Cd, Cr, Pb); Indicator Parameters (TOC, pH, specific conductance).

SHA2.0 SAMPLE COLLECTION

Six different types of samples will be collected: surface soil, surface water, sediment, subsurface soil, subsurface vapor, and groundwater.

SHA2.1 Surface Soils

Surface soil samples include those taken from the surface to a depth of 1.0 ft. In general, any grass or vegetation in the top few inches of soil will be removed from the sample and discarded. The sample will be taken with a small hand held auger or trowel and placed in a pint glass jar. A sheet of aluminum foil will be placed between the jar and lid to provide a seal. A split sample will be taken of all soil samples so that the sample for chemical analyses will remain sealed until the analyses are performed and a separate sample can be used for screening purposes. The sampler will be decontaminated between sample locations.

SHA2.2 Surface Water and Sediment

Surface water and sediment samples will be collected from the tidally influenced drainage ditches and marsh areas during low-tide periods. Surface water samples will be taken with a submersible bottle sampler used to collect water from the entire water column. The sample containers, containing the appropriate preservatives, will be filled directly from the bottle sampler. Sediment samples will be collected using an Ekman dredge or a thin-walled tube-type sampler. All sampling equipment will be decontaminated between sampling locations.

SHA2.3 Subsurface Soils

Subsurface (deeper than 1.0 ft) soil samples will be obtained with either a hand- or gas-powered auger (for shallow samples) or a truck- or buggy-mounted drilling rig (for deeper samples). Samples from the hand equipment will be collected from the blade. The deeper samples will be obtained by hydraulically advancing either a 3-in.-diameter thin-walled tube (cohesive soils) or driving a 2.5-in.-diameter split-spoon sampler (noncohesive soils).

Where possible, dry augering techniques will be used. In general, unless noncohesive soils are encountered, all drilling of borings with completion

depths of 25 ft or less will be completed with dry augering techniques. If necessary, wet rotary methods will be used for advancing deeper boreholes. All samplers will be decontaminated each time they are used and all downhole equipment will be decontaminated between boring locations. Sample cuttings from borings will be contained in drums and held onsite until chemical analyses of the soil have been completed.

After completion of drilling and sampling, each boring will be grouted to the surface from the bottom up. Because of its density, the grout will purge the boring of drilling fluid. The borehole will be grouted from the bottom up using a tremie pipe or by pumping grout through the drill pipe.

The grout mix will consist of 5 to 10 gallons of water per sack (94 lb) of Portland Type I cement and up to 8 lbs of bentonite.

SHA2.4 Subsurface Vapor

Subsurface vapor samples will be obtained by hydraulically advancing a probe which has been designed to collect vapor samples. Soil vapor samples will be collected at 3 ft intervals to a completion depth near the water table. The total volatile organic content (in ppm) in the soil vapor will be determined using a Foxboro Model 128 Organic Vapor Analyzer (OVA). In general, a field gas chromatograph (GC) will be run on vapor samples with OVA readings that exceed 10 ppm to "fingerprint" the contaminants present. Following each sample, the GC column will be backflushed to clear any residual organic vapors.

Due to their small diameter (1/2 in.) and shallow depth (above the groundwater table), holes made while pushing the vadose zone vapor probe and cone penetrometer will not be grouted.

SHA2.5 Groundwater

SHA2.5.1 Monitoring Well Development and Sampling. After groundwater level measurements are made, the well will be developed. Monitoring well installation procedures are included in Section 4.0 of the Work Plan. Development will be performed using a compressed air method or a bottom-filling bailer. The volume of water removed during development will equal a minimum of three times the volume of water standing in the well casing plus approximately three times the volume of any water lost during well installation.

For wells set in low permeable material, when it is not possible to remove these quantities of water, the well will be evacuated to dryness and allowed to recover before sampling. It is planned that samples will be taken within 24 hours of well development. Wells installed and developed during the investigation will be evacuated prior to sampling if more than 24 hours have elapsed since well development. A minimum of three times the volume of water standing in the well casing will be purged. Bailing will be used to obtain water to fill appropriate sample containers. The bailer and any other equipment that contacts the water will be decontaminated between wells. Well development water will be contained in drums onsite, until an appropriate disposal method is determined.

SHA2.5.2 Wellpoint Development and Sampling. The wellpoints will be developed and sampled in the same manner as the monitoring wells.

SHA3.0 DECONTAMINATION

There are two phases of decontamination: equipment and personnel. The equipment decontamination is necessary to reduce the risk of cross contamination during the site investigation and migration of contaminants off site. Personnel decontamination is necessary to protect the workers' health and safety, and is described in detail in the Health and Safety Plan.

The basic format for decontaminating drill pipe, samplers, and tools is a washing and rinsing with detergent and potable water. This procedure will be used during drilling and sampling, before drilling into an uncontaminated strata, and before moving to the next boring location. This decontamination procedure is performed using a 5 percent trisodium phosphate (TSP) solution and a potable water rinse.

Decontamination of the drill rig, bits, samplers, drill pipe, and tools will be completed at each site. If well screens, casing, and centralizers do not arrive from the supplier in factory-sealed packages, the well materials will be decontaminated using a wash of 5 percent TSP and a potable water rinse. Wash solutions will be transferred to drums and disposed of in the same manner as the well development water.

SHA4.0 SAMPLE HANDLING AND ANALYSES

The sample handling procedures will be determined by the types of chemical analyses which are assigned to the sample. The analyses planned for each sample type and sample location are presented in Table SHA1-1.

SHA4.1 Soil and Sediment Samples

All soil and sediment samples will be placed in clean pint jars, covered with foil, sealed with the lid, labeled, and recorded in the field log book. Soil samples collected for organic chemical analyses will be immediately placed in an ice chest and kept at less than 4°C until they are received by the laboratory.

Screening of soil samples will consist primarily of sampling the head space with an organic vapor analyzer (OVA). The sample to be screened will be placed in a pint jar, covered with foil and a lid, and allowed to stand for a period of at least two minutes. The lid will then be removed and the foil pierced with the OVA probe to obtain a measurement of the organic vapor concentration in the headspace above the sample. Samples used for screening will be duplicates of those stored for potential chemical analyses.

Table SHA4-1 summarizes the extraction and analytical methods that will be used during laboratory analysis of the soil and sediment samples.

SHA4.2 Water Samples

Table SHA4-2 summarizes the sample containers and preservatives required for groundwater and surface water samples. Also shown are the analytical methods that will be used during laboratory analysis of the samples.

SHA4.3 Chain-of-Custody Procedures

The purpose of the chain-of-custody (COC) procedures are to provide documentation of the handling of each sample from collection to disposal. This ensures the integrity of the samples for both technical and legal purposes.

Initiation. The COC procedure will be initiated at the site by the Team Leader. As the samples are collected, the Team Leader will:

- assign a unique sample identification number to the sample;
- complete the sample label and affix it to the sample container;

- o take any required field measurements and add preservative (if needed);
- o seal the container with the COC seal (samples for chemical testing only);
- o record the information in a bound field notebook; and
- o place the sample in an ice chest.

Prior to shipment, the COC form will be completed for all the samples being transported.

Sample Identification Numbers. Sample identification numbers will be assigned by the on-site Team Leader. The numbers will be unique to each sample and descriptive. Each identification number will include the date and sample location information.

In general, soil samples will be identified by the location and depth. Groundwater samples will be identified by the monitoring well number and time of collection.

Quality assurance samples (the duplicates and blanks specified in SHA4-3) will be assigned a sample identification number so the laboratory cannot distinguish them from the other field samples.

Sample Labels. After the sample identification number has been assigned, the Team Leader will complete a sample label and affix it to the sample container. An example label is shown in Figure SHA4-1. The labels are self-adhesive and will be completed using a black, waterproof marker.

Chain-of-Custody Seal. After field measurements are made and any preservatives added, the Team Leader will seal the sample container with a tamper-evident seal. An example seal is shown in Figure SHA4-1. For small diameter containers, the seal will be placed around the circumference of the container at the interface of the lid and bottle. For wide-mouthed containers the seal will be placed across the contact between jar and lid. The seals are self-adhesive and will be completed using a black, waterproof marker.

Sample Log Book. All samples obtained for chemical testing will be logged into a field notebook by the Team Leader. The field notebook will be a bound book with consecutively numbered pages and will document the destination of the samples. The Team Leader will be responsible for the field notebook.

Chain-of-Custody Record. An example of a COC form is shown in Figure SHA4-2. This form will be completed by the Field Supervisor prior to securing the samples in an ice chest for transport to the laboratory. After the

samples have been received by the laboratory and inspected, the COC form will be signed and a copy returned to McClelland Engineers. The original will remain with the samples until they are discarded. The method of disposal and date will be noted on the form by laboratory personnel and the form will be returned to McClelland Engineers.

Shipping. The Team Leader will place the COC forms in plastic bags in the ice chests containing the listed samples. The samples will be packed with ice and packing material, if required, to prevent breakage and each ice chest secured with strapping tape. Ice chests containing samples for chemical testing will be transported by overnight carrier or McClelland personnel to the laboratory within 24 hours of being taken. The receipt from the carrier, when used, will be kept by the Team Leader and returned to McClelland as part of the COC records.

Laboratory. It is expected that General Engineering Laboratories, Charleston, South Carolina, will be conducting the laboratory analyses of the samples collected at this activity. When the shipment is received by the laboratory, the ice chests and the individual samples will be inspected for evidence of tampering. Next, the laboratory will verify that the samples received correspond to the samples listed on the COC form. Laboratory personnel will then sign the form and return a copy of it to McClelland Engineers. The original will remain with the samples until they are discarded. McClelland will keep the forms a part of the project file.

McClelland engineers
6100 Hillcroft/Houston, TX 77081

Job No. :
Sample I.D. :
Location :
Sample Type : S SI L G
Preservative : Refrig Acid Base
Sampler : Date :
Comments :

GOF-8.00 (5/85)

McClelland engineers
6100 Hillcroft/Houston, TX 77081

ATTENTION:
Before opening
note if bottle
was tampered with

Sample I.D. :
Sampler : Date :

FIGURE SHA 4-1. EXAMPLE OF CHAIN-OF-CUSTODY LABEL AND SEAL

McCLELLAND ENGINEERS

CHAIN OF CUSTODY RECORD

McClelland engineers

JOB NO	PROJECT NAME	LOCATION

Sample Identification	Date	Time	Sample Container (Size/Material)	Sample Type (Liquid, Soil, etc.)	Preservative	Analyses Requested						Comments
						A	B	C	D	E	F	

Relinquished By (Signature)	Date	Time	Received By (Signature)	Analyses:
				A
				B
				C
				D
				E
				F

Form ENG-1.14 (12/84)

FIGURE SHA 4-2. EXAMPLE OF CHAIN-OF-CUSTODY FORM

Table 4-1
Summary of Sampling, Preservation, and Analysis
Soil Samples

Parameter	Extraction Methods	Analytical Method	Sample Container	Preservation
Volatiles	5030[b]	8240[b]	Pint glass jar with foil-lined lid	Cool <4°C
Semivolatiles	3550[b]	8270[b]	(one sample jar for all analyses)	
Pesticides/PCBs	3540 or 3550[b]	8270[b]		
Metals (Sb, As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	3050[b]	Section 7000[b]		
Chromium, hexavalent				
Cyanides		335.2 or 335[a]		
Total Organic Halogens (TOX)		9020[b]		
Oil and Grease		413.1[a]		

[a] From "Methods for Chemical Analysis of Water and Wastes," EPA, 1979.
[b] From "Test Methods for Evaluating Solid Waste," EPA SW-846, July 1982.

Table 4-2
Summary of Sampling, Preservation, and Analysis
Water Samples

Parameter	Analytical Methods	Sample Container	Preservation
Volatiles	601, 624[a]	Two 40 ml glass VOA vials with Teflon-lined septa. No headspace	No bubbles, pack in inverted position, Cool <4°C
Acid Extractables Base/Neutral Extractables Pesticides/PCBs Dioxins	625[a] 625[a] 608[a] 8250[d]	Half-gallon amber glass bottle with Teflon-lined lid	Cool <4°C
Metals (Sb, As, Ba, Be, Cd, Cr, Cu, Fe, Pb, Hg, Mn, Ni, Se, Ag, Zn)	Section 200[b]	250 ml plastic bottle	Filter on site, HNO_3 to pH <2, Cool <4°C
Cyanide	335.2 or 335.3[b]	500 ml plastic bottle	NaOH to pH >12, Cool <4°C
Phenols, Total	420.1[b]	500 ml glass bottle	H_3PO_4 to pH <4, 0.5 g $CuSO_4$, Cool <4°C
Nitrates	352.1[b]	250 ml plastic bottle	H_2SO_4 to pH <2, Cool <4°C
Total Organic Carbon	415.1[b]	250 ml plastic bottle	HCl to pH <2, Cool <4°C
Chloride Chromium, hexavalent Salinity Sulfate Total Dissolved Solids	407A, 407B[c] 218.4[b] 210[c] 426C[b] 160.1[b]	One quart plastic bottle	Cool <4°C

Table 4-2 Continued

Parameter	Analytical Methods	Sample Container	Preservation
Total Organic Halogens	9020[d]	500 ml amber glass bottle No headspace	Cool <4°C
pH	150.1[b]	250 ml plastic	Determined on site
Temperature			
Specific Conductance	120.1[b]		
Gross Beta Activity	900[d]	Half-gallon plastic bottle with Teflon-lined lid	Cool <4°C
Gross Alpha Activity	900[d]		

a From "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater," EPA.
b From "Methods for Chemical Analysis of Water and Wastes," EPA, 1979.
c From "Standard Methods for the Examinataion of Water and Wastewater," American Public Health Association, 14th Edition.
d From "Test Methods for Evaluating Solid Waste," EPA SW-846, July 1982.
e From "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.

SHA5.0 QUALITY ASSURANCE/QUALITY CONTROL PROGRAM

The purpose of the Quality Assurance/Quality Control (QA/QC) Program is to assess and document the precision and accuracy of the data obtained during the field investigation. The specific objective is to collect data which are of a known quality with respect to:

- precision;
- accuracy;
- completeness;
- representativeness; and
- comparability.

Successful fulfillment of these objectives will require that sound QA/QC practices be observed throughout all phases of the field investigation. The program consists of two parts: (1) the internal (laboratory) quality control, and (2) the external quality assurance. The internal program is documented in "Quality Control in Environmental Testing and Hazardous Waste," prepared by General Engineering Laboratories, Charleston, South Carolina. In this section the external quality control measures are described. In addition to proper sample collection, handling, and documentation which have been described in the previous sections, the Quality Assurance (QA) program includes the submission of QA samples to the laboratory. The samples are sent so that the laboratory will not know they are for QA purposes.

QA samples will be submitted to the laboratory as a check on their internal quality control. QA samples will include field blanks and duplicates. The Team Leader will be responsible for preparing the QA samples and will assign each a number similar to those used for the field samples.

The QA samples are listed in Table SHA5-1. McClelland Engineers will review the results of the QA sample analyses and the documented QC results. If there are apparent inconsistencies, corrective action will be initiated. This will include a review of the field notebook to determine if there were any problems in the sample collection or handling. Next, the laboratory manager will be contacted. The manager will review the laboratory records, and if no reasonable explanation is found and a sufficient quantity of the sample is available, it will be reanalyzed. Documentation of the QA/QC result and any corrective action will become a part of the permanent project file.

Table SHA5-1

Quality Assurance Samples

For MCRD Parris Island

Sample Type	QA Sample	Analyses	Frequency
Water	Duplicate	All	1 in 20
	Blank	Volatiles Only	1 in 20
Soil/Sediment	Duplicate	All	1 in 20

APPENDIX A

QUALITY CONTROL IN ENVIRONMENTAL TESTING AND HAZARDOUS WASTE CHARACTERIZATION, GENERAL ENGINEERING LABORATORIES, CHARLESTON, SOUTH CAROLINA

QUALITY CONTROL
IN ENVIRONMENTAL TESTING
AND HAZARDOUS WASTE CHARACTERIZATION

GENERAL DESCRIPTIONS AND METHODOLOGIES

General Engineering Laboratories
1313 Ashley River Road
Charleston, South Carolina

Volume I

SECTION I

MANAGEMENT STATEMENTS

I. MANAGEMENT STATEMENTS

A. INTRODUCTION

The analytical laboratory provides both quantitative and qualitative data which is used for decision making in many areas. These decisions are based on the protection of human health and environmental resources and often involve the expenditure of large amounts of money. In environmental data, false positive data can result in needless expenditures while false negative data can result in the spread of contamination and serious endangerment to both human health and the environment. Incorrect or misleading data can do more harm than no data at all. The confidence with which one can propose a technical solution to any problem is directly related to the reliability of the information that is available to define the problem.

Data from the laboratory is often used to define whether or not regulatory or contractual conditions are being met. In the case of water, the data is often used to determine whether or not the water can be used for its intended purpose. Decisions involving process changes, plant modifications, or the construction of new facilities may be based primarily on the results of laboratory analyses. The financial implications of such decisions are generally significant and the potential for adverse impact on the environment and the possibility for endangerment to our health is serious. Thus it is essential that the results of analyses conducted in our laboratory be accurate and reliable.

The professional reputation of General Engineering Laboratories is based largely on the accuracy, reliability and prompt turnaround of analytical results from the laboratory. The integrity of the analysts responsible for the analyses and the data clerks responsible for preparing reports and maintaining records must be beyond reproach.

Environmental samples must be tested in accordance with accepted regulatory methods. Standard laboratory procedures must be defined and followed for all aspects of the operation of the laboratory from the preparation of sample bottles and collection of the samples to the final analytical report and archiving of the data. Only Environmental Protection Agency (EPA) methodologies or EPA approved methodologies are used at General Engineering Laboratories. The Standard Operating Procedures (SOP) are documented in Volume I (General and Inorganic Chemistry) and Volume II (Organic Chemistry) of the procedures manuals.

It is essential that all analyses be carried out in strict accordance with the standard procedures in order to insure uniformity of both intralaboratory and interlaboratory test results. It is also essential that all raw data, chromatograms, instrument printouts, and analytical conditions be either recorded in the standard format in the appropriate laboratory data book or be filed in the appropriate laboratory file in order to insure that the test results can be recalled or reconstructed at a later date.

B. STATEMENT OF CORPORATE POLICY FOR QUALITY ASSURANCE AND QUALITY CONTROL

The corporate policy of General Engineering Laboratories for Quality Assurance and Quality Control is threefold:

1: To develop the best technical solution to our client's problem, working always within the constraints of applicable regulations.

2: To present that solution to the client and to the regulatory agencies in a manner which will optimize the probability of acceptance.

3: To accomplish objectives 1 and 2 in the most cost effective manner.

In no case should it be assumed or construed that the management of this company encourages, supports, or in any way condones the execution of any project task in a manner which does not insure that the objectives of the corporate policy will be met.

General Engineering Laboratories has a strong management structure that is dedicated to the generation and reporting of data of known quality. The management requires that every effort be made to insure the accuracy, precision, completeness, and of all results from analyses of environmental samples through the use of stringent Quality Assurance (QA) and Quality Control (QC) procedures. These QA/QC procedures will insure that the integrity of the sample is maintained as the sample passes through each of the following steps of evaluation:

- Collection in the field
- Chemical preservation
- Initiation of Chain of Custody procedures and documentation
- Delivery to the laboratory
- Login
- Storage under appropriate security and temperature conditions
- Analysis by approved methodologies
- Review of analytical data
- Preparation and submission of Certificate of Analysis
- Archiving of data
- Archiving of sample
- Disposal of sample

The company will provide a modern facility with state of the art instrumentation for these analyses. Only qualified and well-trained professionals will be responsible for the results obtained in these analyses.

All analyses will be performed using the latest and most reliable EPA approved methodologies. Quality Control requirements outlined in these procedures will be strictly followed. Only the procedures referenced in the QA/QC Manual will be followed. Any changes in procedures will be updated and submitted to all regulatory and certification agencies that need to be notified prior to implementation of these procedural changes on actual samples. Any changes to the organizational structure will also be submitted to all regulatory and certification agencies when needed. Finally, documentation of any additions of instruments will be submitted to the regulatory and certification agencies with a full Quality Control Startup for that instrument before data is generated from that instrument for regulatory or client use.

C. COMPANY ORGANIZATION AND RESPONSIBILITY

The company organizational chart for General Engineering Laboratories is shown on the following page. This chart represents a flow diagram of the chain of command and responsibility for all sections of the company.

Molly Greene and George Greene, P.E.,Ph.D., are joint owners of General Engineering Laboratories. As President, Molly Greene has ultimate responsibility for the administrative division of the company. This includes client relations, office management,

and accounts receivable and payable. Supervisors of these individual sections report directly to Molly Greene.

As Vice President, George Greene has ultimate responsibility for the technical division of the company. Supervisors and coordinators for the following sections report directly to George Greene: Laboratory Services, Quality Control, Special Projects, Engineering Services, Hydrogeological Services, and Field Services.

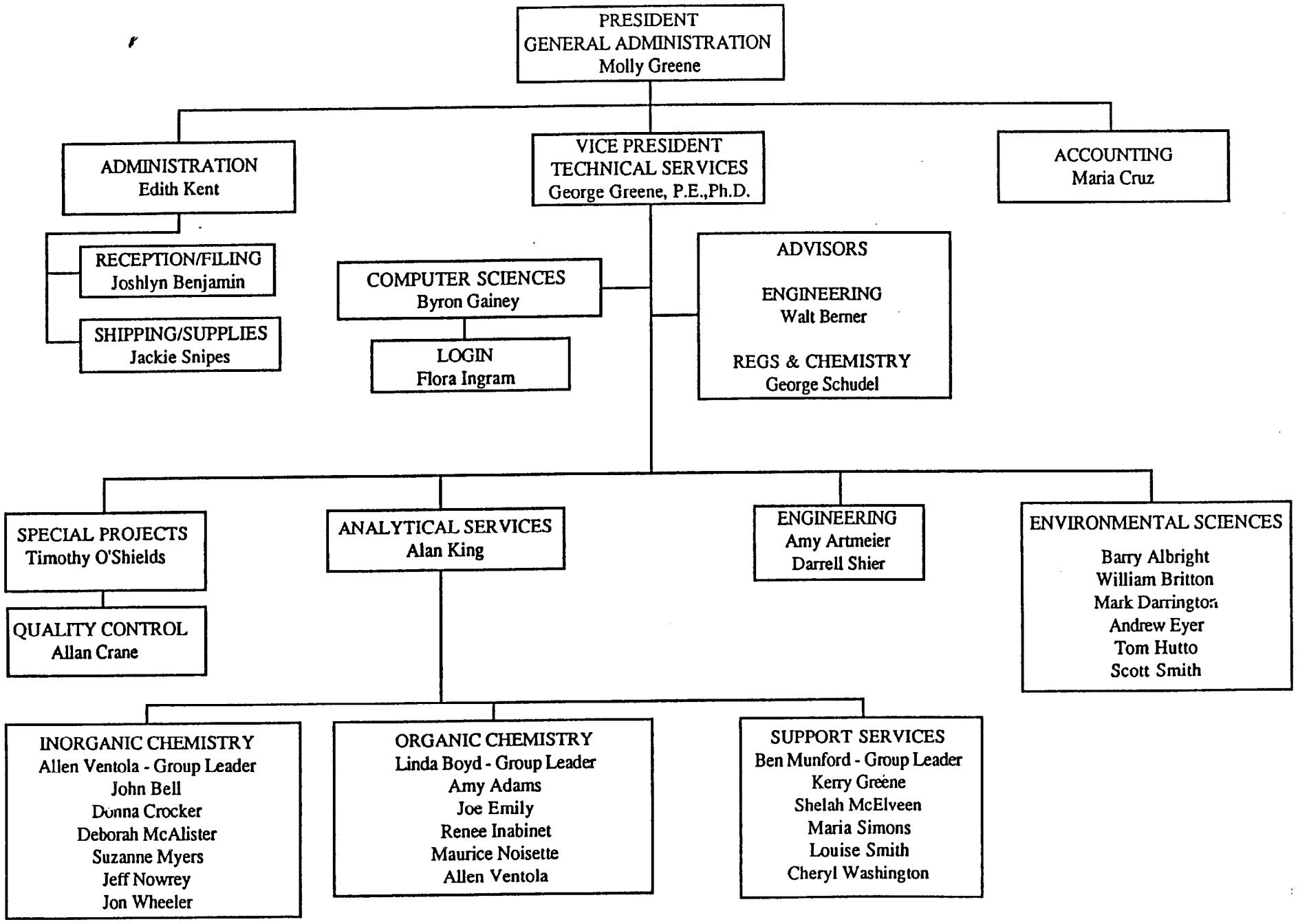
Resumes for each of the professionals in the organization are included in Section XVI of this manual for Quality Control. These resumes provide summaries of the specific duties performed by each individual, his qualifications, including formal education as well as special training and experience related to the employee's specific function in the company.

In the event of an emergency during the weekend or during a second or third shift, the supervisor of the department should be contacted. The following list provides home telephone numbers for supervisors of General Engineering Laboratories:

Greene, Molly 763-1968
Greene, George,P.E.,Ph.D.....763-1968
King, Alan 766-6778
O'Shields, Timothy 762-0761
Shirley, Patrick 571-0026

If an instrument should fail, this emergency situation should be reported as soon as possible to the supervisor of the section. This must be reported immediately if it affects a high volume or fast turnaround requirement. If a principal instrument is not operating, the samples for this instrument must be processed on alternate in-house instruments. General Engineering Laboratories has sufficient redundancy of instrumentation that use of alternate instrumentation for emergency situations presents no problems. It is believed by the management of General Engineering Laboratories that it is more desirable to have in-house redundancy of instrumentation than it is to send work to another laboratory in the event of instrument failure. When samples are sent to another facility, our laboratory loses complete control of the sample, the analytical procedure, and the quality control associated with that sample.

GENERAL ENGINEERING LABORATORIES
AUGUST 1987



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GENERAL ENGINEERING LABORATORIES • 1313 Ashley River Rd • Charleston, SC 29407 • (803) 556-8171

SECTION II

LABORATORY FACILITY AND EQUIPMENT

II. LABORATORY FACILITY AND EQUIPMENT

A. DESCRIPTION OF FACILITIES

General Engineering Laboratories is a complete environmental services company incorporated in Charleston, South Carolina, in 1981, by Molly and George Greene to provide analytical and engineering consulting services to the chemical industry in the area. Since 1981, as a result of the quality and integrity of our professional services and the commitment we have to our clients, the company has grown from a staff of two to a staff of thirty-eight chemists, engineers, geologists, environmental specialists, and technicians. Today we serve industrial clients throughout the southeast as well as other analytical laboratories throughout South Carolina, North Carolina, and Florida, and as far away as Mississippi, Pennsylvania, and Connecticut. Our laboratory is completely equipped with the full complement of state of the art analytical instrumentation required to cost effectively serve our clients' environmental and quality control needs. Computerized data handling procedures allow us to do statistical evaluations and trending studies and provide prompt turnaround. Our chemists and technicians have been specifically trained in the demanding quality control and analytical procedures which are unique to today's state of the art environmental laboratory. Our professional staff provides interpretation of complex analytical data and develops solutions to technical problems within the constraints of the regulatory requirements..

The main laboratory and offices occupy 13,000 square feet in two adjacent buildings on Ashley River Road in Charleston. The laboratory facilities have been specifically designed to meet the stringent quality control and utility requirements of the modern environmental laboratory. The air conditioning system filters return air through electrostatic precipitators to prevent airborne contamination of environmental samples requiring trace level analyses with part per billion detection limits. Analytical instruments are isolated in individual laboratories to insure precise temperature control and to further minimize the possibility of cross contamination. Distillation and extraction procedures are also isolated in separate laboratory rooms to insure quarantine of potentially high concentrations of contaminants. The partition walls in the laboratory are constructed of wire reinforced glass to provide high visibility and a safe working environment for the chemists and technicians. An extensive list of utilities is provided at each work station in the laboratory to insure that analytical procedures can be carried out efficiently and in the most cost effective manner.

In addition to the main laboratory facilities, our company has set up and operated several special purpose laboratories at clients' facilities including mobile laboratories at hazardous waste cleanup sites.

B. UTILITY SERVICES

The following utilities are provided to each individual laboratory and to the individual work stations in the main laboratory by way of pipe racks which run above the ceiling:

+ Cold Water
+ Hot Water
+ Deionized Water
+ Compressed Air

+ Natural Gas
+ Vacuum
+ 110 Volt AC
+ 220 Volt AC (at selected stations)
+ Lighting At 100 foot candles
+ Wastewater Drainage

The pipe racks have been designed to allow utilities to be provided to additional or temporary work stations at any location in the laboratory by simply teeing into the desired overhead line and dropping down to the location.

Cold Water

City water is distributed throughout the laboratory through 3/4 inch Copper tubing. The water enters the building through the Sample Storage Room.

Hot Water

Hot water is distributed throughout the laboratory through 3/4 inch Copper tubing. A 50 gallon quick recovery electric hot water heater is located in the Utility Room.

Deionized Water

Deionized water is distributed throughout the laboratory through half inch schedule 40 PVC pipe. This water is made from city water by a Continental Services Deionization System capable of producing 20 gpm of laboratory pure water. The system is located in the Utility Room and consists of the following units:

+ 10 micron Prefilter: for removal of particulates prior to passing through the ion exchange resin beds;
+ Primary Ion Exchange Bed: for primary removal of trace level cations and anions;
+ Polishing Ion Exchange Bed: for secondary removal of trace level cations and anions and for reserve exchange capacity when the primary cylinder is depleted;
+ Activated Carbon Filter: for removal of trace level organics;
+ Ultraviolet Light: for destruction of any bacterial growth which might pass through the system.

This system is designed to produce laboratory pure water with the following specifications:

+ Conductivity	<5.0 umhos/cm @ 25°C
+ pH	5.5 - 7.5
+ TOC	<1.0 mg/L
+ Trace Metals, Single	<0.05 mg/L
+ Trace Metals, Combined (Cd, Cr, Cu, Ni, Pb, Zn)	<1.0 mg/L
+ Free Chlorine	<0.1 mg/L
+ Standard Plate Count	<10,000/ml

Actual experience and routine monthly Quality Control checks on this water demonstrate that the actual levels of contaminants are below the method detection limits for trace level metals and organics analyses. This water is therefore suitable for use as a blank in all laboratory procedures.

Wastewater Drainage

Wastewater lines run in the service trenches located along the outside walls of instrument rooms and beneath the work benches in the main laboratory. These lines are constructed of two inch schedule 40 PVC pipe to facilitate the construction of additional tie ins and traps when required.

Compressed Air

Compressed air is provided at 70 psig by a Sears two cylinder air compressor, Model No. 106.17554 having a capacity of 5.8 scfm at 90 psi. The compressed air is passed through two Wilkerson filters, Model Nos. F20-02-000 and M20-02-F00, containing activated carbon cartridges to remove trace levels of oil and particulates and distributed throughout the laboratory through half inch schedule 40 galvanized pipe. The compressor and filters are located in the Utility Room.

Natural Gas

Natural gas is distributed throughout the laboratory through half inch schedule 40 black iron pipe for use in applications requiring gas burners. All gas valves have been installed in locations at least three feet removed from electrical outlets and electric radiant heaters.

Vacuum

Vacuum is distributed throughout the laboratory through half inch schedule 40 PVC pipe from a Precision Scientific Model 75 vacuum pump. The vacuum pump is located in the Utility Room. Two small vacuum pumps are available for use at temporary work stations.

Electrical Service - 110 Volt

Electrical service is provided throughout the laboratory for laboratory instrumentation and equipment. All 110 volt outlets are on 20 amp circuits. Computers and laboratory instruments are on dedicated circuits.

Electrical Service - 220 Volt

Several instruments and ovens require 220 volt power. Where required, 30 amp single phase and two phase circuits are provided. Laboratory instruments are on dedicated circuits.

Lighting

Fluorescent lighting is provided throughout the laboratory at a minimum intensity of 100 foot candles at the surface of the work bench. The intensity can be reduced in laboratories with computer terminals to minimize eye strain when working with the CRT.

C. EQUIPMENT

This section discusses specifications, operation, and preventive maintenance requirements of the various pieces of support equipment found in the laboratory. Proper

operation of the laboratory equipment is essential to efficient and reliable operation of the laboratory. Routine maintenance and care of the equipment will minimize down time and help to insure that quality control is maintained in the laboratory.

Examples of Log Sheets used for recording the performance of the various pieces of equipment in the laboratory are shown in Appendix 13.

Autoclaves

The autoclave is used for sterilizing bottles and glassware for bacteriological work. The proper use, care, and maintenance of the autoclave is outlined in the following guidelines:

+ Record the date, time, temperature, type of material sterilized, and analyst's name on the Autoclave Log Sheet each time the autoclave is used.

+ Test the performance of the autoclave with spore strips or suspensions each week.

+ Drain the reservoir after each use and insure that the drain does not become plugged with paraffin or other foreign material.

+ Wipe down the inside and outside each week with a damp cloth.

Automatic Pipets

Automatic pipets are available for general use in the laboratory. These instruments are very precise, however their accuracy must be checked and adjusted before each use. They are particularly useful for preparing standard reagents and for making repetitive dilutions. The proper use, care, and maintenance of the automatic pipets is outlined in the following guidelines:

+ Always store the pipet in an upright position. Never lay the pipet on its side as any moisture in the barrel can then contact the plunger and cause corrosion. The pipet will then become contaminated and lose calibration.

+ Plastic tips should be returned to the wash station for cleaning unless they are badly soiled in which case they should be discarded.

+ Remove the barrel after use and check for signs of liquid in the barrel or on the plunger. Clean thoroughly with deionized water, wipe dry and protect the end of the plunger with a thin film of stop cock grease before reassembling.

Balances

The analytical balances are sensitive instruments capable of detecting the weight of a finger print. They are subject to corrosion from laboratory reagents, finger prints, humidity, and dust. These instruments are located conveniently throughout the laboratory in areas free from drafts and traffic. The benches upon which the balances are placed are designed to be free of vibrations.

The following analytical balances are available for use by trained analysts.

Balance	Location	Number Available
Sartorius Model 1872	Main Laboratory	1
Sartorius Model U6180	Inogainc Prep Lab	1
Mettler Model H78AR	Organic Prep Lab	1
Sartorius Model 1872	GC Lab	1
Mettler	TOX Lab	1

All of the analytical balances are cleaned and adjusted semi-annually by professional technicians under a full service contract. The technician verifies and certifies the accuracy of all analytical during his semiannual visit. Proper daily checks are performed on our balances with Class "S" weights. Balances are verified to be in proper working order and this is documented in a log book for the balances.

Proper use, care, and maintenance of the analytical balances is outlined in the following guidelines:

+ Verify calibration daily with Class "S" weights.

+ Never move a mechanical balance unless the weight and beams have been properly secured.

+ Insure that the balance is level prior to use.

+ Never touch weights, balance pans, or items which are being weighed on the analytical balance with bare hands.

+ Protect all weights, spatulas, and balances from corrosion by keeping the area around the balances clean and free of clutter. Wipe up spills as soon as they occur.

+ Where available, preweigh positions should be utilized to protect the balance from overweight conditions.

+ When not in use, the beam of a mechanical balance should be raised from the knife edge and the weights should be returned to the beam. Objects should be removed from the pans, and the weighing compartment should be cleaned and closed.

+ Return reagents to the reagent shelves and discard weighing boats and papers after use. Insure that the balance benches are clean.

Computers

Computers and peripheral equipment such as terminals, plotters, printers, and tape drives are used throughout the laboratory to log and track samples, monitor the status of laboratory tests, control instruments, evaluate accuracy and precision of laboratory results, and prepare client reports. The computer is extremely integral to the daily operation of the laboratory. It is essential that all laboratory personnel be thoroughly trained in the proper use of the computer and that the computer be properly cared for and maintained. The proper use, care, and maintenance of the computer and peripheral equipment is outlined in the following guidelines:

+ Do not attempt to use the computer without first reading the appropriate manuals and receiving instructions and authorization from your supervisor.

+ Exert care not to damage disks and tapes.

+ Insure that backup disks and tapes are stored in the designated location.

+ Wipe down the computer, peripheral equipment and work area weekly with a damp cloth or as often as required to eliminate the accumulation of dust.

+ Cover computing equipment with a dust cover each evening.

+ Do not place food or drinks near computing equipment.

Distillation Apparatus

The following glass stills are available for distillation procedures:

+ Rotary Vacuum Evaporator: for solvent recovery and procedures requiring low temperature distillation;

+ Cyanide Still: For distillation and absorption of Cyanides as required in the laboratory procedures manual for Cyanide analyses;

+ Soxhlet Extraction Apparatus: for Soxhlet Extraction procedures and other sample prep procedures requiring solvent extractions;

+ General Purpose Reflux and Distillation Apparatus: for special purpose and custom distillation procedures.

Distillation procedures are generally set up and conducted in the Organic Prep Lab. Cooling water, vacuum, and heat are provided to various stations in this room.

Because glass distillation equipment is subjected to extremes in temperatures, special care should be taken minimize thermal stress which could cause the glassware to crack.

Distillation flasks should never be allowed to boil dry any longer than absolutely necessary. Application of heat to a dry flask will bake residues onto the walls of the flask which are difficult if not impossible to remove. High heat on a dry flask can also cause the flask to shatter.

All distillation glassware should be cleaned immediately after use and either set up for future use or placed in the proper glassware drawers.

Filtration Apparatus

Membrane filtration equipment is used for both sterile and nonsterile work. Filtration apparatus used for sterile work should be segregated from other equipment. The proper use, care, and maintenance of the filtration apparatus is outlined in the following guidelines:

+ Use prepackaged sterilized funnels and filters for bacteriological work.

+ Check funnel bodies and bases for scratches on the inside surfaces and signs of leaks.

+ Clean filtration apparatus after each use according to the appropriate glassware cleaning procedures.

Fume Hoods

The following fume hoods are available in the laboratory:

Description	Location	Number	Use
6' fiberglass add-air	Inorganic Prep Lab	1	Corrosives
4' fiberglass	Inorganic Prep Lab	1	Corrosives
6' fiberglass add-air	Organic Prep Lab	1	Organic Extractions
8' fiberglass	Organic Prep Lab	1	Organic Extractions

Each hood is on it's own exhaust system. Make up air for the two add air hoods is provided by a single squirrel cage blower located on the roof above the acid digestion room.

Spills and residues in fume hoods should be wiped up immediately. All fume hoods should be thoroughly wiped down on a weekly basis with a clean damp cloth to prevent the buildup of corrosive or toxic residues.

Fume hoods are checked by a certified industrial hygienist on a regularly scheduled quarterly basis. The fume hoods are checked for proper face velocities (100 FPM), for proper exhaust capabilities, and for any signs of malfunction. These quarterly checks are documented in a log book for fume hoods. Signs of corrosion or malfunction should be noted and scheduled for repair as soon as possible.

Grinders and Homogenizers

The grinder is located in the acid digestion room. Insure that the wheels are not adjusted tighter than necessary in order to minimize unnecessary wear on the wheels. Flush the grinder with an ample portion of the sample before collecting the aliquot for analysis. Allow the grinder to run until all of the sample has been run through but do not allow the grinder to run empty longer than necessary.

Hot Plates and Heating Mantles

Hot plates and heating mantles are available to provide heat for distillations, digestions, and sample concentrating procedures.

Care should be taken to protect heating mantles from spills which could deteriorate the insulating materials and cause electrical shorts.

The proper use, care, and maintenance of the hot plates and heating mantles is outlined in the following guidelines:

+ Inspect electrical components regularly for signs of wear or corrosion. Equipment which shows signs of possible electrical malfunction should be tagged and scheduled for immediate repair.

+ Wipe down hot plates used for acid digestions after each use with a damp cloth to minimize corrosion.

+ Remove hot plates and variacs from corrosive environments when not in use.

Incubators

Several incubators are available for bacteriological work and Biochemical Oxygen Demand (BOD) analyses. The proper use, care, and maintenance of the incubators is outlined in the following guidelines:

+ The Total Coliform Incubator should be maintained at 35±0.5°C. Measure the temperature daily with a thermometer having 0.5°C divisions and record the temperature on the Total Coliform Incubator Log Sheet.

+ The Fecal Coliform Incubator should be maintained at 44.5±0.2°C. Measure the temperature daily with a thermometer having 0.1°C divisions and record the temperature in the Fecal Coliform Incubator Log Sheet.

+ The BOD Incubator should be maintained at 20±1°C. Measure the temperature daily with a thermometer having 1°C divisions and record the temperature in the BOD Incubator Log Sheet.

+ Wipe the incubators down inside and outside with a damp towel once a month.

+ Drain the water from the Fecal Coliform Incubator monthly, clean with detergent and refill.

Microscopes

One phase contrast and one binocular dissecting microscope are available for microscopic examinations. These microscopes are located in the bacteriology laboratory. The phase contrast microscope should be used only by trained analysts. The proper use, care, and maintenance of the microscopes is outlined in the following guidelines:

+ Clean the optics and stage after each use with lens paper.

+ Keep the instrument covered when not in use.

+ Store optics in the proper container.

Muffle Furnaces

Several muffle furnaces are available for ashing samples and for other procedures requiring high temperatures. Muffle furnaces should be used in the Inorganic Prep Lab. If a smokey sample is to be ashed, the muffle furnace should be placed in a fume hood. The proper use, care, and maintenance of the muffle furnaces is outlined in the following guidelines:

+ Inspect electrical components regularly for signs of wear or corrosion. Equipment which shows signs of possible electrical malfunction should be tagged and scheduled for immediate repair.

+ Wipe down the inside and outside of the muffle furnaces each week with a damp cloth to minimize corrosion.

Ovens

Ovens are available at various locations in the laboratory. Several ovens are for general purpose drying. The temperature settings of these ovens may be adjusted as needed for the particular application. Other ovens are used for special purpose analyses. The temperature settings on these ovens should not be adjusted unless the ovens are out of calibration. The proper use, care, and maintenance of the ovens is outlined in the following guidelines:

+ The Residues Drying Oven should be set at 105±1°C. Measure the temperature daily with a thermometer having 0.5°C divisions and record the temperature on the Residues Drying Oven Log Sheet.

+ Clean up spills immediately with a damp cloth.

+ Wipe down the inside and outside of the ovens monthly with a damp cloth.

+ Ovens must have a thermometer in a medium (sand, etc.) to monitor the temperature of that oven. The temperature must be checked on a regular basis and must be recorded on a temperature check chart that must be posted on the outside of the oven.

Quebec Colony Counter

The Quebec Colony Counter is used for identifying and counting bacteria colonies. The proper use, care, and maintenance of the colony counter is outlined in the following guidelines:

+ Wipe down the magnifying lens and light stand with a soft damp cloth to insure that they are clean and free of dust particles prior to use.

Refrigerators

Several refrigerators are available for storage of samples, standards, and reagents which are subject to degradation at ambient temperatures. The proper use, care, and maintenance of the refrigerators is outlined in the following guidelines:

+ Sample storage refrigerators should be set at 4°C. Measure the temperature daily with a thermometer having 0.5°C divisions and record the temperature on the Sample Refrigerator Log Sheet.

+ Identify and date all containers in the refrigerator and remove and discard outdated material.

+ Clean up spills immediately with a damp cloth.

+ Wipe down the inside and outside of the refrigerators monthly with a damp cloth.

Thermometers

Thermometers are used throughout the laboratory in most analyses to obtain support data for the primary information being sought. It is important that the proper thermometer be selected for the specific task at hand to insure accurate results. The proper use, care, and maintenance of the thermometers is outlined in the following guidelines:

+ Insure that the proper thermometer is being used for the particular application. Select a thermometer with the appropriate immersion length and sufficient temperature range and divisions to provide the accuracy required.

+ In many cases thermometers are dedicated to a specific use. Care should be taken to protect these thermometers from breaking.

+ After use, clean the thermometer with detergent and cold water. Place it back in its protective case and return it to the thermometer drawer.

+ All thermometers must be verified and calibrated against a National Bureau of Standards (NBS) Thermometer before use and periodically thereafter to verify that the thermometer is maintaining its calibration. A written record must be maintained in the thermometer files to document these NBS thermometer checks.

Ultraviolet Sterilizer

The ultraviolet sterilizer is required for bacteriological work. The proper use, care, and maintenance of the sterilizer is outlined in the following guidelines:

+ Unplug the unit and clean the UV lamps monthly by wiping with a clean soft cloth moistened with ethanol.

+ Perform spread plate irradiation quarterly to insure that the lamps are operating efficiently.

+ Test the UV lamps quarterly with a light meter and replace if they emit less than 80% of their rated output.

Water Baths

Several water baths are available for a variety of procedures in various locations in the laboratory. The proper use, care, and maintenance of the water baths is outlined in the following guidelines:

+ Clean up spills immediately with a damp cloth.

+ Drain the water monthly and clean the inside of the bath with detergent.

+ Wipe down the outside monthly with a damp cloth.

+ A temperature and maintenance record must kept for each water bath that is used by the laboratory. Temperature charts must be recorded on each water bath on a daily basis on each day that the water bath is used. Active charts should be posted at each water bath. Completed charts should be maintained in the water bath maintenance files.

D. INSTRUMENTATION

Accurate and reliable analysis in the laboratory is insured by the use of EPA approved analytical procedures and state of the art instrumentation. Training sessions are held regularly to familiarize the analysts with new methodologies. Proper use and care of the instrumentation in the analytical laboratory is essential for the development of accurate and reliable data. The following discussion of analytical instrumentation is organized into the three areas of analyses: Metals, Organics, and General Chemical.

METALS ANALYSES

Metals analyses are done in the laboratory on advanced Perkin Elmer instrumentation using the following technologies:

Inductively Coupled Plasma (ICP)

The Perkin Elmer Plasma II ICP is used for most metals analyses. This technology generally gives better detection limits than conventional flame atomic absorption technology and is subject to fewer interferences. Instrument software allows analyses to be made at multiple emission wavelengths to avoid interferences and to confirm results. The emission spectra can be examined by the spectroscopist when the instrument indicates the potential presence of interferences. In this way the spectroscopist can determine whether or not interfering spectral lines are giving false positives. The importance of this capability can not be overemphasized when the potential economic impact of a false positive is considered.

Zeeman Furnace

The Zeeman Z-3030 Furnace is also used for low level metals analyses when detection limits below those obtainable by ICP are required. With this furnace technology, it is essential that the analytical method be optimized for each sample matrix. The instrument software allows the spectroscopist to set temperatures and instrument conditions while observing the absorption spectra. In doing so, matrix interferences which this technology are typically subject to can be minimized, and the method parameters can be optimized. The Zeeman Furnace techniques generally provide better methods for reducing matrix interferences than the conventional furnace techniques.

Flame Atomic Absorption (AA)

The Perkin Elmer Model 2380 AA is used to analyze for certain metals (such as Sodium and Lithium) whose emission/absorption spectra are more stable at the lower temperatures of conventional flame AA. Here also, the spectra can be examined by the spectroscopist when interferences are suspected. This instrument has the capability to use either air/Acetylene or Nitrous Oxide/Acetylene flames for analysis.

Cold Vapor Atomic Absorption

The spectrometer and data processing sections of the Perkin Elmer Model 2380 AA are used with an Instrumentation Laboratories Atomic Vapor Accessory Model 440 for routine and low level Mercury analyses.

Graphite Furnace (HGA)

The Perkin Elmer Model HGA 500 is used for low level metals analyses when detection limits below those obtainable by ICP are required. Typically, this technology is required for drinking water and groundwater analyses. With this technology, it is essential that the analytical method be optimized for each sample matrix. The instrument software allows the spectroscopist to set temperatures and instrument conditions while observing the absorption spectra. In doing so, matrix interferences which this technology are typically subject to can be minimized, and the method parameters can be optimized.

ORGANICS ANALYSES

Routine as well as custom organics analyses are done in the laboratory on state of the art Tracor Model 560 gas chromatographs, Tracor Model 540 gas chromatographs, Perkin Elmer Sigma 300 gas chromatograph, a Finnigan Model 1050 H GC/Mass Spectrometer, and a Finnigan Model 5100 EF/H GC/Mass Spectrometer. Screening type analyses are done for indicators of organic contamination on a Dohrmann Model DC 80 Total Organic Carbon Analyzer and a Mitsubishi Model 10 Total Organic Halide Analyzer.

Output from all of the Gas Chromatographs is fed directly into the Minicomputer by way of dual channel interfaces. The analyst has the ability to monitor instrument performance and manipulate raw data from the computer console. Specialty software integrates the raw data, identifies peaks, regresses the data, and interfaces the computed results with the results data base for the sample under analysis.

Tracor Model 560 GC

The gas chromatographs are equipped with the following detectors and accessories:

+ Electron Capture Detector (ECD): The Electron Capture Detector is used for most analyses requiring the detection of low levels of organic compounds in water samples. This detector is typically capable of achieving detection limits in the fractional part per billion range. Special training and licenses are required for this instrument because of the radioactive isotope in the detector.

+ Hall Electrolytic Conductivity Detector (Hall): The two Hall Electrolytic Conductivity Detectors are used for analyses of organic compounds containing Halogens, Nitrogen, or Sulfur groups. These detectors can also be used for the analysis of volatile organics in water samples, as well as for the analyses of extracts of soils, sludges, and oils. They are also used to confirm results obtained on the ECD.

+ Flame Ionization Detector (FID): The Flame Ionization Detector is used for routine analyses of wastewaters and hazardous wastes. This instrument is capable of detecting a broad spectrum of organic compounds, however, it is generally not capable of achieving the detection limits obtainable with the ECD and Hall Detectors.

Both 560 gas chromatographs are equipped with both packed and capillary columns. Capillary columns are used in analyses requiring high resolution and low detection limits. Packed columns are used for confirmation and custom organic analyses. One of the 560 gas chromatographs is equipped with dual electron capture detectors and

two different columns for reliable confirmations of quantitated parameters using simultaneous two column confirmations.

Tracor Model 540 GC

The gas chromatographs are equipped with the following detectors and accessories:

+ Photoionization Detector (PID): The Photoionization Detector is used for routine analyses of water and waste waters for aromatic compounds. The PID is used in sequence with a Hall Electrolytic Conductivity Detector to detect the volatile fraction of the Priority Pollutant Constituents.

+ Hall Electrolytic Conductivity Detector (HECD) configured in halogen mode: The Hall Detector configured in halogen mode is used to detect halogenated organics in all environmental matrices. The HECD is a very specific detector, but it is also a very sensitive detector. With proper sample introduction, an analyst can easily reach part per billion detection limits for most compounds. The HECD is used in sequence with a PID to detect the full volatile fraction of the Priority Pollutant Constituents.

+ Electron Capture Detector (ECD): The Electron Capture Detector is used for most analyses requiring the detection of low levels of organic compounds in water samples. This detector is typically capable of achieving detection limits in the fractional part per billion range. Special training and licenses are required for this instrument because of the radioactive isotope in the detector.

+ All three 540 Gas Chromatographs are equipped with both capillary and packed column injectors.

+ Two 540 Gas Chromatographs are equipped with Tekmar Purge and Trap Systems and ten-place automatic samplers. The automatic samplers allow samples to be analyzed overnight unattended. The two instruments are dedicated to the analysis of priority pollutant volatiles using EPA Method 601 and 602. These two instruments are equipped with sequential photoionization detector and Hall electrolytic conductivity detector in halogen mode.

Perkin Elmer Sigma 300 GC

This gas chromatograph is equipped with the following detectors and accessories:

+ Flame Ionization Detector (FID): The Flame Ionization Detector is used for routine analyses of wastewaters and hazardous wastes. This instrument is capable of detecting a broad spectrum of organic compounds, however, it is generally not capable of achieving the detection limits obtainable with the ECD and Hall Detectors. The flame ionization detector is commonly used for the analysis of high level organics, such as the EPA "F" listed compounds.

+ Thermal Conductivity Detector (TCD): The Thermal Conductivity Detector is used for routine analyses of water and waste waters. The TCD is commonly referred to as a "hot wire" detector. In the TCD, heat is carried by the carrier gas from an electrically heated hot wire to the wall of the detector cell. For best sensitivity, the carrier gas should be either helium or hydrogen (the sensitivity with nitrogen is about 6% of that with hydrogen, and the peaks go off in both directions from the baseline). As with any GC detector, the TCD should be

kept hot enough to prevent condensation of sample. In practice, this means that the detector is kept about 25°C hotter than the highest anticipated column temperature. Both maximum sensitivity and maximum detector life dictate, however, that TCD detector temperature should be no higher than necessary. The cooler the heated element can be run, the longer it will last. Thus, not only should the cell temperature be minimized, but the current heating the element should be held to the minimum that will provide the necessary sensitivity for the analysis. The attenuation should be set at a fairly low level to take advantage of most of the detector signal. The Thermal Conductivity Detector can detect any substance that can be eluted from a GC column. Thus, the TCD is useable for most gas chromatographic analyses.

+ Perkin Elmer HS-6 Headspace Sampler: The Perkin Elmer HS-6 Headspace Sampler is an automatic sampling attachment that is used for the direct analysis of solid waste and sludge samples for volatile organics. Liquid samples may also be analyzed by this method, but the purge and trap method is more likely to be the method of choice for liquid samples. This sampler is typically used on a gas chromatograph equipped with a flame ionization detector and a thermal conductivity detector for analysis of high level volatile organics.

Finnigan Model 1050 H GC/Mass Spectrometer

This GC/Mass Spec is used for Priority Pollutant analyses, Appendix VIII, and Appendix IX analyses, and comprehensive analysis of unknown organic compounds. This instrument is one of the most versatile GC/Mass Spec available today for the identification of unknown organic compounds. The instrument is equipped with both packed and capillary columns and uses state of the art technology and a computer data base of over 42,000 organic compounds to identify and quantitate unknown organics. Strict quality control procedures are built into the operation of this instrument to confirm the analytical results and eliminate false positives commonly found in environmental samples.

The Finnigan Model 1050 H GC/ Mass Spectrometer/ Data System is primarily used for the analysis of volatile organics. It is equipped with a Tekmar LSC-2 purge and trap assembly and a Tekmar ALS automatic sampler.

This Finnigan 1050 H has the following standard features:

- Electron ionization source
- 800 u mass analyzer
- Perkin Elmer Sigma 300 temperature programmable gas chromatograph
- Dual purpose, capillary and packed column injector
- Split/splitless operation in capillary mode
- Capillary transfer line direct into the ion source
- Turbomolecular-pumped vacuum system
- 16-bit minicomputer with 64 megabytes of memory
- 70-megabyte Winchester-type disk drive
- 20-megabyte streaming tape drive
- High resolution video display terminal with detachable keyboard
- 150 IBM Printer/Plotter
- NBS/EPA library of mass spectra of more than 42,000 compounds
- State of the art Super Incos software
- AutoQuan software for automatic batch processing
- Packed column operation with glass jet separator
- Oscilloscope monitor

- High-speed serial data-communications interface with the operating software called Data Master
- Q/A Formaster™ automatic EPA CLP forms generation

Finnigan Model 5100 EF/H GC/Mass Spectrometer

This GC/Mass Spec is used for Priority Pollutant analyses, Appendix VIII, and Appendix IX analyses, and comprehensive analysis of unknown organic compounds. This instrument is one of the most versatile GC/Mass Spec available today for the identification of unknown organic compounds. The instrument is equipped with both packed and capillary columns and uses state of the art technology and a computer data base of over 42,000 organic compounds to identify and quantitate unknown organics. Strict quality control procedures are built into the operation of this instrument to confirm the analytical results and eliminate false positives commonly found in environmental samples.

The Finnigan Model 5100 EF/H GC/ Mass Spectrometer/ Data System is primarily used for the analysis of semi-volatile extractable organics, including the base/neutral and acid extractables. It is equipped with a carousel-type automatic sampler that will hold up to 60 concentrated extracts for automatic processing.

This Finnigan 5100 EF/H has the following standard features:

- Electron ionization source
- 800 u mass analyzer
- Finnigan Model 9611 temperature programmable gas chromatograph
- Dual purpose, capillary and packed column injector
- Split/splitless operation in capillary mode
- Capillary transfer line direct into the ion source
- Turbomolecular-pumped vacuum system
- 16-bit minicomputer with 64 megabytes of memory
- 70-megabyte Winchester-type disk drive
- 20-megabyte streaming tape drive
- High resolution video display terminal with detachable keyboard
- 150 IBM Printer/Plotter
- NBS/EPA library of mass spectra of more than 42,000 compounds
- State of the art Super Incos software
- AutoQuan software for automatic batch processing
- Packed column operation with glass jet separator
- Oscilloscope monitor
- High-speed serial data-communications interface with the operating software called Data Master
- Q/A Formaster™ automatic EPA CLP forms generation

Dohrmann Model DC 80 TOC Analyzer

The TOC analyzer is used for the determination of Total Organic Carbon (TOC) as well as Total Carbon in aqueous samples. TOC is one of the indicator parameters required for routine groundwater monitoring by state and federal regulations. Accurate and reproducible analysis is critical in any groundwater program. This instrument utilizes ultraviolet light, Potassium Persulfate, and Ultra High Purity Oxygen to oxidize the organic compounds to Carbon Dioxide which is then quantitated in an infrared detector. This state of the art technology eliminates most of the maintenance and stability problems common with the older high temperature combustion technologies. Peripheral equipment allow this

instrument to be used for the determination of TOC in solids and sludges, as well as for the determination of Purgeable Organic Carbon in aqueous samples.

Mitsubishi Model 10 TOX Analyzer

The TOX analyzer is used for the determination of Total Organic Halogens (TOX) in both aqueous and nonaqueous samples. TOX is another one of the groundwater indicator parameters required by federal and state regulations. As with TOC, accurate and reproducible analysis is critical in any routine groundwater program. This instrument utilizes the absorption, combustion, and coulometric titration technologies specified in EPA Method 9020. The microprocessor controlled titration allows rapid recovery of the instrument and a high degree of precision.

GENERAL CHEMISTRY ANALYSES

Wet chemical analyses are done with a variety of speciality and general purpose instruments. The following discussion presents these instruments in alphabetical order.

Conductivity Meters

Several portable conductivity meters are available for both laboratory and field use. The proper use, care, and maintenance of the conductivity meters is outlined in the following guidelines:

+ Check the cell constants periodically to insure that the meter conditions have not changed.

+ If drifting or erratic response is experienced, check the batteries and replace if required.

+ Because Conductivity is a function of temperature, it is important to either measure the Conductivity at 25°C or record the temperature at the time of measurement.

+ Rinse the probe with deionized water after use and replace the protective cover. Badly soiled probes may require a detergent wash prior to the deionized water rinse.

Selective Ion and pH Meters

Selective ion and pH meters are available for use at several locations in the laboratory. Portable meters may also be used for field work.

Plastic bodied, gel filled combination pH electrodes have been found to give the most reliable service and are therefore used for all routine laboratory work.

The proper use, care, and maintenance of these meters is outlined in the following guidelines:

+ Calibrate the pH meters in accordance with the procedure applicable to the meter. Record the date, standard readings, expiration date of the standard, and the analyst's name on the pH Meter Calibration Log Sheet.

+ Date the standard buffers when they are prepared and discard them on or before the expiration date.

+ Because pH is a function of the temperature, it is important to either measure the pH at 25°C or to record the temperature at the time of measurement.

+ Rinse the probe with deionized water after use and either replace the protective cover or store the electrode in the appropriate standard solution.

+ Wipe down the instrument weekly with a damp cloth.

RFA 300

The Alpkem RFA 300 (Rapid Flow Analyzer) is used for the quantitation of total phenols in aqueous samples. It is also routinely used for the analysis of samples for nitrate and nitrite. This instrument is completely automated to facilitate the analysis of quality control spikes and blanks. Other wet methods such as Cyanides, and Nutrients can be easily handled by this instrument with the insertion of additional modules.

Spectrophotometer - UV/Visible

The spectrophotometer is available for colorimetric analyses in the laboratory. The proper use, care, and maintenance of this instrument is outlined in the following guidelines:

+ Wipe down the surface of the instrument weekly with a damp cloth.

+ Carefully clean the filters and lens weekly with lens paper.

+ Replace the light source when the instrument response becomes unstable.

+ Optical cells should be handled with extreme care to prevent scratches. The cells should be acid cleaned if badly soiled. Care should be taken when cleaning with a brush to prevent scratching. Finger prints and smudges must be removed with lens paper before inserting the cell into the instrument.

+ Light absorbance will change as the optical cells are rotated from one position to another. It is therefore essential to insure that the cells are always inserted into the instrument with the mark in exactly the same position.

+ Instrument sensitivity should be monitored against standard solutions to detect the occurrence of electrical, mechanical, or optical problems at an early stage.

Turbidimeter

The turbidimeter is used for the determination of turbidity in the laboratory. The proper use, care, and maintenance of this instrument is outlined in the following guidelines:

+ Calibrate the instrument on the day of use with standard Formazin solution.

+ Protect the sample cuvettes from scratches and fingerprints.

+ Wipe the instrument down weekly with a damp cloth.

E. GLASSWARE

The measurement of constituents in water and wastewater at the ppm and ppb levels demands methods of maximum sensitivity. These methods are very sensitive to contamination resulting from improperly cleaned glassware. Thus the task of cleaning glassware given given as much attention by the analyst as the test procedure itself.

Several types of glassware and glassware related items are used in the laboratory:

+ Borosilicate or polyethylene: These materials can be used for all analyses, sample collections, and reagent storage unless instructed otherwise by the specific procedure.

+ Kimax and Pyrex: These are trade names for conventional heat resistant borosilicate glass. Both are used interchangeably in the laboratory. Almost all of the volumetric glassware as well as beakers, and cylinders are made of these materials.

+ Corex: This material is harder and more expensive than conventional borosilicate glass. Most of the pipets in the inorganic laboratory are made of this material in order to minimize breakage.

+ Glass or high density polyethylene stoppers are used with ground glass joints. Cork or rubber stoppers are used in other cases, however, rubber is not used when organic analyses are to be performed.

+ Teflon plugs are used in burets and teflon stopcocks are used in separatory funnels.

+ Teflon liners are used in sample bottles for routine water and wastewater analyses.

+ Aluminum foil can be used as cap liners for organics analyses in lieu of Teflon.

VOLUMETRIC GLASSWARE

Class A volumetric glassware must be used for all quantitative analytical work. Solutions must be measured at the temperature at which the glassware was calibrated (usually 20° C).

Volumetric flasks are calibrated "to contain" (TC).

Volumetric pipets are calibrated "to deliver" (TD). When emptying volumetric pipets, they should be held in the vertical position and the outflow should be unrestricted. The tip of the pipet should be put in contact with the wall of the receiving vessel for approximately two seconds after the free flow has stopped. In no case should the remaining fluid be blown or tapped from the tip of the pipet.

Burets are used to deliver definite volumes. Burets should not be dried after cleaning. Rather they should be rinsed two or three times and left filled with the titrant. If the buret is not to be used for a period of time it should be filled with deionized water. All burets should be capped or covered with aluminum foil to prevent dust from accumulating on the inside. The stopcock, if not Teflon, should be greased with a very thin film of stopcock grease to prevent it from freezing up.

Chipped or cracked volumetric glassware should not be used for quantitative work unless the calibration is unaffected by the damage.

CLEANING OF GENERAL GLASSWARE

All glassware for general wet chemistry procedures should be cleaned according to the following steps:

+ Rinse well with tap water.

+ Wash or soak in a 5% solution of Contrad. Contrad is a phosphate free, biodegradable surface active cleaning agent obtained from Curtin Matheson Scientific Company. The Contrad is diluted to 5% by volume. The 5% solution is good as long as the pH is > 8.5.

+ Rinse thoroughly with tap water.

+ Rinse with three small volumes of deionized water.

+ Badly soiled glassware is cleaned by the above procedure and then soaked in a Chromic Acid bath for fifteen to thirty minutes. Sometimes, an overnight soaking is required to remove organic residues.

+ Rinse glassware soaked in Chromic Acid thoroughly with both tap water and deionized water to remove any levels of chromium.

+ Note that Chromic Acid is extremely corrosive. Wear protective clothing when working with this material and use caution to prevent spills.

CLEANING OF ORGANIC GLASSWARE

Cleaning of glassware for organics analyses requires a solvent rinse and a period of time to dry in a drying oven. The following steps should be carefully followed to insure that the glassware is scrupulously clean:

+ Rinse the glassware with tap water immediately after use.

+ If the glass is extremely dirty, it may be soaked overnight in a water bath.

+ Wash the glass in the Contrad solution described in the procedure for cleaning general laboratory glass. Liquinox may be substituted for Contrad.

+ Rinse thoroughly with hot tap water.

+ Rinse thoroughly with deionized water.

+ Rinse thoroughly with nanograde hexane.

+ Rinse thoroughly with nanograde acetone.

+ Place in a drying oven that is at 180 to 200°C for at least one hour.

+ One exception to the above glass preparation is the prep of forty milliliter vials for the analysis of volatile organics. These vials are placed in the oven after the deionized water rinse. No solvent is used to rinse this glassware. Also, these vials should not be placed in the oven with any solvent-rinsed glass. Cross contamination can easily occur when solvent-rinsed glassware is mixed in the oven with volatile organic analysis glassware.

+ Glassware for herbicide analysis is rinsed with 10% HCl before the acetone rinse.

CLEANING OF GLASSWARE FOR METALS ANALYSIS

Erlenmeyer flasks used in metals analysis and BOD bottles used for mercury analysis are cleaned according to the following procedure:

+ 150 ml of the following acid mixture is boiled in the Erlenmeyer or BOD bottle until it flushes against the entire surface area of the glass. (The acid mixture is 600 ml of deionized water + 200 ml of nitric acid + 200 ml of hydrochloric acid + 50 ml of sulfuric acid.)

+ Rinse the acid-washed glass thoroughly with deionized water.

+ Volumetric flasks, graduated cylinders, pipets, and other glass used in metal analyses are cleaned in the following procedure:

+ Rinse the glassware thoroughly with 2 N nitric acid.

+ Rinse the acid-rinsed glassware thoroughly with deionized water.

F. REAGENTS, SOLVENTS, AND GASES

Analytical Reagent Grade (AR) chemicals are used for most analyses in the inorganic laboratory. Ultrapure or pesticide grade reagents are used for organic work. This includes pesticide grade solvents.
If the purity of a reagent is not specified by the procedure, it is intended that AR Grade chemicals be used.

Standards and solutions should be labeled with the following information when they are prepared:

+ Compound
+ Concentration
+ Parameters for which reagent is to be used
+ Date
+ Name of Analyst

Also when a standard is prepared, it should be logged into the Standard Log Book. There is a separate Standard Log Book for organic standards and general wet tests/metals standards. The standard should be listed in the overall log at the beginning of the book. Specific weighing data and other critical information should be included in the Stock Standard Section of the book. If separate intermediate and working standards are prepared from the stock standard, then they should be included in the Sections marked Intermediate Standards and Working Standards in the Log Book. In these sections are also included

other important information about the preparation and storage of these standards. When a standard is out of date or discarded, it should be deleted from the current standard log book. The Standard Log Book should be reviewed periodically. Standard Methods gives the shelf life for the inorganic and wet chemistry standards and reagents. Pesticide and herbicide stock standards should be prepared once a year and intermediate and working standards should be prepared twice a year. Volatile organic standards should be prepared on a working basis. If volatile organic stock standards are stored, they should be stored in very small vials with no head space. Even with proper storage, volatile standards do not last more then one to two weeks. Refer to Section X under Laboratory Quality Control Checks for more details on the procurement and documentation for standards.

INORGANIC ANALYSES

AR grade reagents are used for all quantitative inorganic analyses.

To avoid waste and to insure that fresh reagents are used for quantitative procedures, only the amount of reagent required for the scheduled work should be prepared.

Zero grade oxygen is used for the TOC analyzer.

METALS ANALYSES

ICP/AA standards are purchased as 1000 ppm stock standards from the various scientific supply houses. Working standards and sample dilutions are prepared with deionized water.

Acid digestions are done with AR grade Nitric and/or Hydrochloric Acid. Samples and standards must be brought to the same acid Normality before analysis.

The following gases, purchased from Sunox or Merrit Holland, are used for ICP/AA work:

+ Liquid Argon
+ Lab 380 Atomic Absorption Acetylene
+ Medical Grade Nitrous Oxide
+ Ultra high purity Nitrogen

Air is supplied to the AA by the laboratory air compressor (see the Utilities Section for details).

ORGANIC ANALYSES

Nanograde or pesticide grade solvents are used for organic glass prep, extraction, and analysis. Standard reference material is purchased from Chem Service or obtained from the EPA repository at Research Triangle Park, North Carolina.

The following gasses are purchased from Sunox or Merrit Holland and are used for Organic work:

+ GC/MS
  - Breathing Quality Air

- Ultra high purity Helium

+ Gas Chromatographs
  - Ultra high purity Helium
  - Ultra high purity Hydrogen
  - Breathing Quality Air
  - Ultra high purity P-10 Mixture of Argon/Methane
  - Reagent Grade Carbon Dioxide for subambient cooling on Tracor 540

+ TOX Analyzer
  - Zero Grade Argon
  - Zero Grade Oxygen

+ TOC Analyzer
  - Zero Grade Oxygen

+ Tennelec Alpha, Beta, and Gamma Counter
  - Ultra high purity P-10 Mixture of Argon/Methane

Gases are passed through drying tubes and molecular sieves before entering the GC.

REAGENT BLANK

A background or reagent blank should be determined for each new lot of solvent or reagent for each procedure. Refer to the specific procedure for details.

SECTION III

QUALITY ASSURANCE OBJECTIVES

III. QA/QC OBJECTIVES

The parameters analyzed by General Engineering Laboratories are divided into three main categories:

- Metals Analyses
- General Inorganic Analyses
- Organic Analyses

For each parameter in these categories, it is essential to establish limits of acceptance for Quality Control data. Therefore, QA/QC objectives for precision, accuracy, and completeness must be established or defined.

The definitions of precision, accuracy, and completeness are as follows:

- Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions."

- Accuracy - The degree of agreement of a measurement (or an average of measurement of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100 (X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias of a system.

- Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.

All measurements must be made such that results are representative of the media (i.e. surface water, groundwater, soil/sediment, solid waste, etc.) and conditions being measured. Unless otherwise specified, all data must be calculated and reported in units consistent with other organizations reporting similar data.

The accuracy and precision control limits for each of the parameters in the three categories for analysis are based on:

- Historical Data
- Method Validation Studies
- Replicate Analyses
- Spike Analyses
- Standard Analyses
- Calibrations
- Recovery Studies

The above criteria were used to determine most of the QA Objectives that are documented in Tables 1, 2, 3, and 4 of this section. Where sufficient data was not available, EPA Method control data limits will be used as QA criteria until inhouse data is available

Table 1
Data Quality Objectives - Metals

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
Aluminium	200.7	-	6.4	100.1	98,n=82
Antimony	200.7	-	17.9	97.3	89,n=54
Arsenic	206.2	-	12.6	93.8	95,n=80
Barium	200.7	-	6.5	101.6	96,n=80
Beryllium	200.7	-	12.9	97.4	96,n=24
Boron	200.7	-	7.7	99.8	94,n=63
Cadmium	200.7	-	6.2	99.0	95,n=79
Calcium	200.7	-	8.0	99.4	98,n=41
Chromium	200.7	-	5.3	100.4	96,n=79
Cobalt	200.7	-	6.9	101.4	99,n=79
Iron	200.7	-	6.8	102.4	94,n=52
Lead	200.7	-	12.9	101.9	97,n=79
Lithium	200.7	-	ID[f]	ID	ID
Magnesium	200.7	-	7.5	97.1	91,n=56
Manganese	200.7	-	8.0	101.9	94,n=84
Mercury	245.1	-	10.1	99.6	94,n=98
Molybdenum	200.7	-	ID	ID	ID
Neodymium	200.7	-	ID	ID	ID
Nickel	200.7	-	9.5	101.0	97,n=101
Niobium	200.7	-	ID	ID	ID
Palladium	ID	-	ID	ID	ID
Phosphorous	200.7	-	6.5	103.0	92,n=12
Potassium	200.7	-	8.4	97.5	96,n=28
Selenium	270.2	-	11.0	96.7	94,n=80
Silicon	200.7	-	2.6	95.8	100,n=10
Silver	200.7	-	8.4	97.6	92,n=101
Sodium	200.7	-	4.6	98.6	92,n=61
Strontium	200.7	-	7.2	104.3	89,n=9
Sulfur	200.7	-	5.9	102.0	83,n=12
Thallium	200.7	-	9.7	92.8	96,n=26
Thorium	200.7	-	ID	ID	ID
Tin	200.7	-	11.6	98.9	86,n=14
Titanium	200.7	-	9.9	97.4	96,n=25
Uranium	200.7	-	ID	ID	ID
Vanadium	200.7	-	13.5	98.0	82,n=11
Zinc	200.7	-	6.5	101.1	93,n=236

a Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March, 1979.
b Spike Concentrations and Matrices as Milligrams of Test Parameter per Liter of Sample where Both are Variable.
c Standard Deviation of Percent Recovery.
d Average Percent Recovery.
e Percent of n Analysis Results within $\pm 2\sigma$.
f ID=Insufficient Data as of This Printing.

Table 2
Data Quality Objectives - General Chemistry

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
Acidity	305.1	-	NA[f]	NA	NA
Alkalinity	310.1	-	NA	NA	NA
BOD	405.1	-	NA	NA	NA
Chloride	325.3	-	8.4	99.6	93,n=83
Chlorine	330.5	-	NA	NA	NA
COD	410.4	-	92.5	92.5	93,n=54
Conductance (Specific)	120.1	-	NA	NA	NA
Corrosivity	203[g]	-	NA	NA	NA
Cyanide	335.2	-	98.6	98.6	88,n=56
Flash Point		-	NA	NA	NA
Fluoride	340.1	-	101.8	101.8	95,n=94
Formaldehyde		-	97.0	97.0	83,n=6
Hardness (Total)	130.2	-	NA	NA	NA
Heating Value		-	NA	NA	NA
Nitrogen (Ammonia)	350.2	-	103.0	103.0	93,n=67
Nitrogen (Kjeldahl)	351.3	-	102	102	92,n=33
Nitrogen (Nitrate)	353.3	-	13.3	90.1	72,n=47
Nitrogen (Nitrite)	353.3	-	11.5	95.6	94,n=17
Odor	140.1	-	NA	NA	NA
Oil&Grease	413.1	-	9.2	91.7	93,n=92
pH		150.1	-	±0.1	99.7
Phenol	420.1	h	7.7	99.6	88,n=58
Phosphorous (Ortho)	365.2	-	10.4	97.9	86,n=35
Phosphorous (Total)	365.3	-	10.4	97.9	86
Reactivity	9010	-	NA	NA	NA
Solids (Dissolved)	160.1	-	NA	NA	NA
Solids (Setteable)	160.5	-	NA	NA	NA
Solids (Suspended)	160.2	-	NA	NA	NA
Solids (Total)	160.3	-	NA	NA	NA
Solids (Volatile)	160.4	-	NA	NA	NA
Sulfate	375.4	-	7.6	95.4	92,n=83
Sulfide	376.2	-	27.6	86.5	76 n=25

Table 2
Data Quality Objectives - General Chemistry

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
(H_2S)					
Sulfite	377.1	-	NA	NA	NA
Surfactants (MBAS)	425.1	-	13.4	90.9	90,n=25
Turbidity	180.1	-	NA	NA	NA
TOC	415.2	-	11.0	94.5	94,n=99
TOX	9020	-	NA	NA	NA

a Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March, 1979.
b Spike Concentrations and Matrices as Milligrams of Test Parameter Per Liter of Sample Where Both are Variable.
c Standard Deviation of Percent Recovery.
d Average Percent Recovery.
e Percent of n Analysis Results within ±2s.
f NA=Not Available, QA Objectives Based on Intralaboratory and Interlaboratory Precision and Accuracy Statement, Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, March, 1979.
g Langlier Index "Standard Methods for the Examination of Water and Waste Water", 14 Edition, 1976.
h Micrograms of Test Parameter Per Liter of Sample.

Table 3
Data Quality Objectives - Organics

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
Acenaphthene	625	100	27.6	60.4-132.8	80-120
	610	100	40.3	D-105.7	80-120
Acenaphthylene	625	100	40.2	53.5-126.0	80-120
	610	100	45.1	22.1-112.1	80-120
Acetone	1624	-	-	-	-
Acrolein	1624	-	-	-	-
	603	50	4.6	42.9-60.1	80-120
Acrylonitrile	1624	-	-	-	-
	603	50	9.9	33.1-69.9	80-120
Anthracene	625	100	32.0	43.4-118.0	80-120
Benzene	624	20	6.9	15.2-26.0	80-120
	602	20	4.1	10.0-27.9	80-120
Benzidine	1625	100	119	16-518	80-120
Benzo(a)anthracene	625	100	27.6	41.8-133.0	80-120
	610	10	4.0	3.1-11.6	80-120
Benzoic Acid	1625	-	-	-	-
Benzo(a)pyrene	625	100	39.0	31.7-148.0	80-120
	610	10	4.0	0.2-11.0	80-120
Benzo(b)fluoranthene	625	100	38.8	42.0-140.4	80-120
	610	10	3.1	1.8-13.8	80-120
Benzo(ghi)perylene	625	100	58.9	D-195.0	80-120
	610	10	2.3	D-10.7	80-120
Benzo(k)fluoranthene	625	100	32.3	25.2-145.7	80-120
	610	5	2.5	0-7.0	80-120
Benzyl Chloride	f	-	-	-	-
Benzyl Butyl Phthalate	625	100	23.4	D-139.9	80-120
	606	10	4.2	5.7-11.0	80-120
Biphenyl	1625	100	41.0	75-148	80-120
Bis(2-chloroethoxy) methane	625	100	34.5	49.2-164.7	80-120
	611	100	25.7	27.3-115.0	80-120
Bis(2-chloroethyl) ether	625	100	55.0	42.9-126.0	80-120
	611	100	26.3	26.3-136.8	80-120
Bis(2-ethylhexyl)phthalate	625	100	41.1	28.9-136.8	80-120
	606	50	38.4	1.2-55.9	80-120
Bromodichloromethane	624	20	6.4	10.1-28.0	80-120
	601	20	4.3	10.7-32.0	80-120
Bromomethane	624	20	17.9	D-41.2	80-120
	601	20	7.6	3.4-24.5	80-120
Bromoform	624	20	5.4	11.4-31.1	80-120
	601	20	4.7	5.0-29.3	80-120
4-Bromophenylphenyl ether	625	100	23.0	64.9-114.4	80-120
	611	100	39.3	7.6-167.5	80-120
Carbazole	1625	100	38	36-165	80-120
Carbon tetrachloride	624	20	5.2	17.2-23.5	80-120
	601	20	5.6	11.8-25.3	80-120

Table 3
Data Quality Objectives - Organics

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
Chloroethane	624	20	11.4	8.4-40.4	80-120
	601	20	4.4	11.3-25.2	80-120
4-Chloro-3-methylphenol	625	100	37.2	40.8-127.9	80-120
	604	100	16.6	56.7-113.4	80-120
Chlorobenzene	624	20	6.3	16.4-27.4	80-120
	601	20	5.0	10.2-27.4	80-120
2-Chloroethylvinyl ether	624	20	25.9	D-50.4	80-120
	601	20	8.3	4.5-35.5	80-120
Chloroform	624	20	6.1	13.7-24.2	80-120
	601	20	4.5	12.4-24.0	80-120
Chloromethane	624	20	19.8	D-45.9	80-120
	601	20	7.4	D-34.9	80-120
2-Chloronaphthalene	625	100	13.0	64.5-113.5	80-120
	612	100	37.3	29.5-126.9	80-120
2-Chlorophenol	625	100	28.7	36.2-120.4	80-120
	604	100	27.0	54.1-110.2	80-120
4-Chlorophenylphenyl ether	625	100	33.4	38.4-144.7	80-120
	611	100	30.7	15.4-152.5	80-120
Chrysene	625	100	48.3	44.1-139.9	80-120
	610	10	4.2	D-17.5	80-120
p-Cymene	1625	100	18	76-140	80-120
n-Decane	1625	100	51	24-195	80-120
Dibenzo(a,h)anthracene	625	100	70.0	D-199.7	80-120
	610	10	2.0	0.3-10.0	80-120
Dibenzofuran	1625	100	20	85-136	80-120
Dibenzothiophene	1625	100	31	79-150	80-120
Dibromochloromethane	624	20	6.1	13.8-26.6	80-120
	601	20	6.3	7.9-35.1	80-120
1,2-Dichlorobenzene	625	100	30.9	48.6-112.0	80-120
	601	20	5.5	1.7-38.9	80-120
1,3-Dichlorobenzene	625	100	41.7	16.7-153.9	80-120
	601	20	9.1	6.2-32.6	80-120
1,4-Dichlorobenzene	625	100	32.1	37.3-105.7	80-120
	601	20	5.5	11.5-25.5	80-120
3,3'-Dichlorobenzidine	625	100	71.4	8.2-212.5	80-120
Dichlorodifluoromethane	601	-	-	-	-
1,1-Dichloroethane	624	20	5.1	14.2-28.5	80-120
	601	20	3.2	11.2-24.6	80-120
1,2-Dichloroethane	624	20	6.0	14.3-27.4	80-120
	601	20	5.2	13.0-26.5	80-120
1,1-Dichloroethene	624	20	9.1	3.7-42.3	80-120
	601	20	6.6	10.2-27.3	80-120
trans-1,2-Dichloroethene	624	20	5.7	13.6-28.5	80-120
	601	20	6.4	11.4-27.1	80-120
2,4-Dichlorophenol	625	100	26.4	52.5-121.7	80-120
	604	100	25.1	59.7-103.3	80-120

Table 3
Data Quality Objectives - Organics

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
1,2-Dichloropropane	624	20	13.8	3.8-36.2	80-120
	601	20	5.2	10.1-29.9	80-120
cis-1,3-Dichloropropene	624	20	15.8	1.0-39.0	80-120
	601	20	7.3	6.2-33.8	80-120
trans-1,3-Dichloropropene	624	20	10.4	7.6-32.4	80-120
	601	20	7.3	6.2-33.8	80-120
Diethyl ether	1624	-	-	-	-
Diethyl phthalate	625	100	26.5	D-100.0	80-120
	606	25	9.0	1.9-33.4	80-120
2,4-Dimethylphenol	625	100	26.1	41.8-109.0	80-120
	604	100	33.3	50.4-100.0	80-120
Dimethyl phthalate	625	100	23.2	0-100.0	80-120
	606	25	9.5	1.3-35.5	80-120
Di-n-butyl phthalate	625	100	16.7	8.4-111.0	80-120
	606	25	8.9	10.3-29.6	80-120
Di-n-octyl phthalate	625	100	31.4	18.6-131.8	80-120
	606	50	13.4	D-50.0	80-120
2,4-Dinitrophenol	625	100	49.8	D-172.9	80-120
	604	100	36.0	31.7-125.1	80-120
2,4-Dinitrotoluene	625	100	21.8	47.5-126.9	80-120
	609	20	5.1	3.6-22.8	80-120
2,6-Dinitrotoluene	625	100	29.6	68.1-136.7	80-120
	609	20	4.8	3.8-23.0	80-120
p-Dioxane	1624	-	-	-	-
Diphenylamine	1625	100	45	58-205	80-120
Diphenyl ether	1625	100	19	82-136	80-120
1,2-Diphenylhydrazine	1625	100	73	49-308	80-120
n-Docosane	1625	100	31	45-152	80-120
n-Dodecane	1625	100	74	35-369	80-120
n-Eicosane	1625	100	59	53-263	80-120
Epichlorohydrin	Note	-	-	-	-
Ethylbenzene	624	20	7.5	17.4-26.7	80-120
	602	20	6.7	10.0-28.2	80-120
Fluoranthene	625	100	32.8	42.9-121.3	80-120
	610	10	3.0	2.7-11.1	80-120
Fluorene	625	100	20.7	71.6-108.4	80-120
	610	100	43.0	D-119	80-120
Hexachlorobenzene	625	100	24.9	7.8-141.5	80-120
	612	10	2.4	2.6-14.8	80-120
Hexachlorobutadiene	625	100	26.3	37.8-102.2	80-120
	612	10	2.2	D-12.7	80-120
Hexachlorocyclopentadiene	625	100	15	69-144	80-120
	612	10	2.5	D-10.4	80-120
Hexachloroethane	625	100	24.5	55.2-100.0	80-120
	612	10	3.3	2.4-12.3	80-120
n-Hexacosane	1625	100	35	35-193	80-120

Table 3
Data Quality Objectives - Organics

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
n-Hexadecane	1625	100	33	80-162	80-120
Hexanoic acid	1625	-	-	-	-
Ideno(1,2,3-cd)pyrene	625	100	44.6	D-150.9	80-120
	610	10	3.0	1.2-10.0	80-120
Isophorone	625	100	63.3	46.6-180.2	80-120
	609	100	32.3	8.0-100.0	80-120
Methylene Chloride	624	20	7.4	D-41.0	80-120
	601	20	4.0	7.0-27.6	80-120
2-Methyl-4,6-Dinitrophenol	625	100	93.2	53.0-100.0	80-120
	604	100	25.0	42.4-123.6	80-120
Methyl ethyl ketone	1624	-	-	-	-
Naphthalene	625	100	30.1	35.6-119.6	80-120
	610	100	40.7	21.5-100.0	80-120
ß-Naphthylamine	1625	100	49	10-NS	80-120
Nitrobenzene	625	100	39.3	54.3-157.6	80-120
	609	100	33.3	25.7-100.0	80-120
2-Nitrophenol	625	100	35.2	45.0-166.7	80-120
	604	100	22.5	56.6-103.8	80-120
4-Nitrophenol	625	100	47.2	13.0-106.5	80-120
	604	100	19.0	22.7-100.0	80-120
N-Nitrosodimethylamine	1625	100	198	21-472	80-120
	607	20	3.4	4.6-20.0	80-120
N-Nitosodi-n-propylamine	625	100	55.4	13.6-197.9	80-120
	607	20	5.7	11.5-26.8	80-120
N-Nitrosodiphenylamine	1625	100	45	65-142	80-120
	607	20	6.1	2.1-24.5	80-120
n-Octacosane	1625	100	35	35-193	80-120
n-Octadecane	1625	100	39	42-131	80-120
2,2-oxybis(1-chloropropane)	625	-	-	-	-
	611	-	-	-	-
PCB-1016	625	-	-	-	-
	608	50	100	30.5-51.5	80-120
PCB-1221	625	-	-	-	-
	608	50	24.4	22.1-75.2	80-120
PCB-1232	625	-	-	-	-
	608	50	17.9	14.0-98.5	80-120
PCB-1242	625	-	-	-	-
	608	50	12.2	24.8-69.6	80-120
PCB-1248	625	-	-	-	-
	608	50	15.9	29.0-70.2	80-120
PCB-1254	625	-	-	-	-
	608	50	13.8	22.2-57.9	80-120
PCB-1260	625	100	54.2	19.3-121.0	80-120
	608	50	10.4	18.7-54.9	80-120
Pentachlorophenol	625	100	48.9	38.1-151.8	80-120
	604	100	32.4	56.7-113.5	80-120

Table 3
Data Quality Objectives - Organics

Parameter	Method[a] (EPA No.)	Matrix[b] (mg/L)	Precision[c] (SD)	Accuracy[d] (X%)	Completeness[e] (%)
Phenanthrene	625	100	20.6	65.2-108.7	80-120
	610	100	37.7	8.4-133.7	80-120
Phenol	625	100	22.6	16.6-100.0	80-120
	604	100	14.1	32.4-100.0	80-120
a-Picoline	1625	100	38	59-149	80-120
Pyrene	625	100	25.2	69.6-100.0	80-120
	610	10	3.4	1.4-12.1	80-120
Styrene	1625	100	42	53-221	80-120
a-Terpineol	1625	100	44	42-234	80-120
2,3,7,8-TCDD[g]	613	0.100	0.0276	0.0523-0.1226	80-120
1,1,2,2-Tetrachloroethane	624	20	7.4	13.5-27.2	80-120
	601	20	9.2	6.6-31.8	80-120
Tetrachloroethene	624	20	5.0	17.0-26.6	80-120
	601	20	4.2	9.2-26.6	80-120
n-tetracosane	1625	100	11	80-139	80-120
n-Tetradecane	1625	100	109	NS-985	80-120
Toluene	1624	20	6.3	14.5-28.7	80-120
	602	20	4.0	11.2-27.7	80-120
n-Triacontane	1625	100	32	61-200	80-120
1,2,3-Trichlorobenzene	1625	100	69	15-229	80-120
1,2,4-Trichlorobenzene	625	100	28.1	57.3-129.2	80-120
	612	100	31.6	20.2-133.7	80-120
1,1,1-Trichloroethane	624	20	4.6	13.7-30.1	80-120
	601	20	4.9	10.8-24.8	80-120
1,1,2-Trichloroethane	624	20	5.5	14.3-27.1	80-120
	601	20	3.9	9.6-25.4	80-120
Trichloroethene	624	20	6.6	18.6-27.6	80-120
	601	20	4.2	9.2-26.6	80-120
Trichlorofluoromethane	601	20	6.0	7.4-28.1	80-120
2,3,6-Trichlorophenol	1625	100	30	58-137	80-120
2,4,5-Trichlorophenol	1625	100	30	58-137	80-120
2,4,6-Trichlorophenol	625	100	31.7	52.4-129.2	80-120
	604	100	16.6	60.8-110.4	80-120
Vinyl Chloride	624	20	20	0-43.5	80-120
	601	20	5.7	8.2-29.9	80-120

a Environmental Protection Agency Approved Methods as Promulgated Under 40 CFR Part 136, Federal Register, Vol. 49, No. 209, Rules and Regulations.
b Unless Otherwise Noted all Spikes are as Micrograms of Test Parameter per Liter of Reagent Water.
c Expected Standard Deviation Limit of Four Test Recovery Measurements in µg/L of Spiked Reagent Water.
d Expected Average Test Recovery of Four Tests in µg/L of Spiked Reagent Water.

Table 3
Data Quality Objectives - Organics

e Average Percent Recovery from Spiked Sample.
f "Selected Analytical Methods Approved and Cited By the United States Environmental Protection", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).
g Tetrachloro-Dibenzo-p-Dioxin.
D = Detected, result must be greater than zero.
NS = No Specification, limit is outside range that can be measured reliably.

SECTION VI

CALIBRATION PROCEDURES AND FREQUENCY

VI. CALIBRATION PROCEDURES AND FREQUENCY

Analyses at General Engineering Laboratories are divided into three major categories of testing:

- Metals Analyses
- General Inorganic Analyses
- Organic Analyses

Calibration procedures vary for the different categories of analysis. Frequency for these calibrations, however, is the same. All instruments are calibrated daily at General Engineering Laboratories unless the EPA Methodologies used for the analysis required more frequent calibration.

Calibration for Metals Analyses

All analyses for metals at General Engineering Laboratories are performed in strict accordance with the methodologies outlined in the EPA 600 Series Analytical Methods and the EPA SW-846 Methods of Analysis. Most analyses are performed using Inductively Coupled Plasma (ICP) techniques. In cases where there are matrix interferences or where detection limits are lower than achievable by ICP methods, Zeeman Furnace or conventional graphite furnace methods are used for analysis. For mercury analyses, the cold vapor technique is used.

For all of these analyses, calibration methods are the same. The steps for calibration are extensively outlined in the EPA Methodologies and in the Perkin Elmer Instrument Manuals for instruments used in these analyses. The Standard Operating Procedure for calibration of the ICP system, the furnaces, or the mercury analyzer are outlined in the General Engineering Laboratories Procedures Manual for Inorganic and General Chemistry.

The ICP is calibrated automatically using the Perkin Elmer ICP software for each element before an analysis for that particular element. This calibration is performed using multi-level standard concentrations to establish calibration curves. The response factor versus this calibration curve for various standards is intermittantly verified using a standard at the reporting level and other non-calculation standards during the course of an analytical run. If the response factor is off ± 5%, the calibration is performed again. After re-calibration, the calibrationis verified with an external standard response factor check.

Similar Perkin Elmer software is used to calibrate the Zeeman furnace and the conventional graphite furnace. Again, if the response factor standard check is not in a ±10% agreement with the calibration curve, the system is re-calibrated and a verification of this calibration documented.

As with the other forms of elemental analyses, the same calibration procedures apply to the analysis of mercury. Again, if the response factor standard check is not in ±10% agreement with the calibration curve, the system must be re-calibrated and a verification of this calibration documented.

Calibration for General Inorganic Chemistry Analyses

All analyses for general inorganic chemistry and wet chemistry parameters at General Engineering Laboratories are performed in strict accordance with the methodologies outlined in the EPA 600 Series Analytical Methods and the EPA SW-846 Methods of

Analysis. Most of these methods are either colorimetric, titrimetric, or instrumental in nature.

For general inorganic chemistry parameters and the analysis of wet chemistry parameters using colorimetric techniques, calibration curves are performed on each day that the test is run. This calibration is accomplished using a minimum of three standards to form the calibration curve. In many instances, up to five standards are used to form this calibration curve. For these tests, the standards are carried through the procedure in the same manner as the samples. The calibration curves for wet and general chemistry are documented in the bound laboratory books on the day that the curve is generated.

For general inorganic chemistry parameters and the analysis of wet chemistry parameters using titrimetric techniques, standardization of the titrant is performed on each day that the test is run. The standardization process is described in detail on a parameter basis in the General Engineering Laboratories Procedures Manual for Inorganic and General Chemistry. The documentation of the standardization process is included in the lab book writeup for the test on the date that the data is generated.

For general inorganic chemistry parameters that are being analyzed using autoanalyzer instruments, calibrations are performed using at least three concentrations of standards within the linear working range of the instrument. These calibrations are performed each day that the instrument is used to generate data.

Calibration for Organic Chemistry Analyses

All analyses for organic chemistry parameters at General Engineering Laboratories are performed in strict accordance with the methodologies outlined in the EPA 600 Series Analytical Methods and the EPA SW-846 Methods of Analysis. These methodologies include analyses using both gas chromatographic techniques (GC) and gas chromatographic/mass spectrometer techniques (GC/MS).

For the organic analyses, the calibration procedures are different for GC/MS work versus GC with other detectors. The EPA Methods give explicit instructions for the calibrations and verification of calibration for these systems used in organic analyses.

Calibration of the GC system requires the analysis of an external standard followed by a response factor check standard. If there is not 80 to 120% agreement between these two sequential standards, corrective action must be taken before any samples or spikes can be analyzed. This type of GC calibration is described in detail in both the General Engineering Laboratories Organic Chemistry Procedures and the following EPA Methods:

- EPA 601 or 8010
- EPA 602 or 8020
- EPA 604 or 8040
- EPA 606 or 8060
- EPA 607 or 8070
- EPA 608 or 8080
- EPA 609 or 8090
- EPA 610 or 8100
- EPA 611 or 8110
- EPA 612 or 8120

These calibration methods are applicable to all the parameters included in the above methods, including volatile organics, pesticides, PCBs, phthalate esters, acid extractables, polynuclear aromatic hydrocarbons, and other base/neutral extractables.

The GC/MS system must be tuned using the specified tuning compounds in the EPA methodologies (DFTPP for extractables and BFB for volatiles - see Section IX). After EPA tuning criteria are met, the system must be calibrated using an instrument contained fluorocarbon compound, FC-43. Again, this calibration check must meet the EPA specified criteria for acceptance before analyses can be performed. After the internal calibration of the GC/MS system using the system FC-43, an external calibration must also be performed and/or verified. A three to five point calibration must be performed for the parameters for analysis. If this three to five point calibration has already been established, a single standard can be analyzed to verify that the calibration is still valid. If the calibration is not within the specified limits that are outlined in the EPA Methods 624/625 (8240/8250), the three to five point calibration must be redone before samples or quality control references can be processed. If the calibration has already been performed, this verification of the calibration must be performed before each work shift when samples are to be processed.

The calibration standards used to perform the instrument calibrations and verifications are obtained from various sources. Calibration Standards are obtained from the EPA Repository of Pesticides and Industrial Chemicals (MD-8), Research Triangle Park, North Carolina 27709 USA. The standards obtained from this facility are in the neat form. The phone number for this facility is (919) 541-3951. The emergency number is (702) 798-2690. This facility will not accept phone orders except in the event of an emergency. They will offer technical assistance for standards and methodologies over the phone. The standards to be ordered are selected from the EPA Document: Analytical Reference Standards and Supplemental Data: The Pesticides and Industrial Chemicals Repository/ EPA-600/4-84-082. A copy of the order blank is added to this QC Manual as Appendix 7.

Pre-weighed Standards at ppm concentrations are also available to our laboratory from the United States Environmental Protection Agency, Quality Assurance Branch, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, 26 West St. Clair Street, Cincinnati, Ohio 45268. The telephone number for this laboratory is (513) 569-7325. This laboratory will accept phone orders for QC Samples that need to be ordered. The order will be processed and delivered to our laboratory within four to six weeks. A record of standards ordered and received must be maintained in the Standard Log Book for both inorganics and organics. A limit of six standards for each parameter are allotted to our laboratory on an annual basis. The most recent standards that are available through this office and the order form for these standards is added to this Quality Control Manual as Appendix 8.

Certified standards are also used by our laboratory from the following sources:

Chem Service Inc.
PO Box 3108
West Chester, Pennsylvania 19381

Supelco Inc.
Supelco Park
Bellefonte, Pennsylvania 16823-0048

Aldrich Chemical Company
940 West Saint Paul Avenue
Department 700
Milwaukee, Wisconsin 53233 USA

Sigma Chemical Company
PO Box 14508
St. Louis, Missouri 63172 USA

American Scientific Products
8350 Arrowbridge Blvd.
Charlotte, North Carolina 28210

Fisher Scientific Company
Department 301276-01
PO Box 405
Pittsburgh, Pennsylvania 15230

Only the highest grade of standards available are purchased for use in our laboratory. The standards, when purchased in the neat form, are certified to have a known purity. If purchased in a pre-weighed or pre-diluted form, the solutions are certified to be of a given concentration. When either neat standards or certified solutions are received in the central supply area, the container is marked as received and dated with the date of that day.

When standards are prepared in stock, intermediate, and working concentrations, either from the neat form or from a certified solution, the preparation of that standard is documented in a bound Standard Log Book. There are separate volumes of the Standard Log Book for:

- Metals
- General Inorganics
- Volatile Organics
- Pesticides/Herbicides/PCBs/Phthalate Esters
- Base/Neutral and Acid Extractables
- General Organics, including Hazardous Waste Characterization Standards for Organic Analyses

The forms used to document the preparation of stock, intermediate, and working standards are added to this QC Manual as Appendix 9. Holding times for standards vary. Expiration dates are given on both the EPA standards and the standards that are purchased from the vendors. No standard is ever used after the expiration date. Standards that are in solution are meniscus-marked and periodically checked to insure that no concentration of the solvent has occurred. If there is evident concentration of the solvent medium, the standard solution is discarded and replaced with a freshly prepared standard solution.

SECTION VII

ANALYTICAL METHODOLOGIES

VII. ANALYTICAL METHODOLOGIES

Analyses are performed at General Engineering Laboratories using only EPA methodologies or EPA approved methodologies. References for methods used by General Engineering Laboratories include EPA 600 Series Methods and Quality Control Requirements for Organic, Inorganic, and General Chemistry, the SW-846 Methods for the analysis of solid waste and groundwater samples, Standard Methods for Analysis of Water and Wastewater, 16th Edition, AOAC Methodologies, and ASTM Methodologies.

Procedures Manuals at General Engineering Laboratories include procedures for every parameter that is tested by the laboratory. These procedures are step-by-step instructions for sample preparation, analysis, instrument calibration, quality control requirements, and reporting of data.

The normally reported detection limits for each parameter analyzed by General Engineering Laboratories included in this section as Tables 1 through 4. The detection limits were determined on a parameter by parameter basis using the guidelines for detection limit determinations outlined in EPA Methods of Chemical Analysis of Water and Wastes/USEPA-600/4-79-020 and Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975.

INORGANIC AND GENERAL CHEMISTRY PROCEDURES

Acidity - EPA Method 305.1/USEPA-600/4-79-020 - This is a titrimetric method that measures the mineral acidity as well as the acidity contributed by the oxidation and hydrolysis of any polyvalent cations which might be present in the sample. This procedure is applicable to water and wastewater including those containing reduced polyvalent cations.

Alkalinity - EPA Method 310.2/USEPA-600/4-79-020 - This is a titrimetric method that is applicable to the determination of alkalinity as $CaCO_3$, in water and wastewater samples.

Ash Percentage Determination - EPA Methods of Chemical Analysis of Water and Wastes/USEPA-600/4-79-020 and Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 208 - This is a gravimetric method for the determination of % Ash in soils and sludges. It indicates a weight loss at 550° C after drying at 100° C. Samples containing volatile solvents or materials which evaporate at 100° C will give a low bias to the results. Non-volatile residue is the remainder of the material after the ash procedure has been completed.

Biochemical Oxygen Demand (BOD) - EPA Method 405.1/USEPA-600/4-79-020 - This method is a five day air incubation and dissolved oxygen determination by membrane electrode. The procedure is applicable to water and wastewater samples. Samples from industrial waste samples that contain toxic materials may require special study and treatment.

Bromide - EPA Method 320.1/USEPA-600/4-79-020 - This method is applicable to drinking, surface, and saline waters, including domestic and industrial waste effluents containing 2 to 20 milligrams of bromide per liter of water. Iron, manganese, and organic matter can interfere with this test; however, the calcium oxide pretreatment removes or reduces these to insignificant concentrations. Color interferes with observations of indicator, but this interference can be overcome by the use of a pH meter instead of a pH indicator and the use of standardized amounts of oxidant and oxidant-quencher.

Chemical Oxygen Demand (COD) - EPA Method 410.4/USEPA-600/4-79-020 - This is a colorimetric procedure for the determination of COD in water and wastewater. Samples, blanks, and standards are sealed in tubes and heated in an oven in the presence of dichromate at 150° C. After 2 hours, the tubes are removed from the oven, cooled, and measured spectrophotometrically at 600 nm.

Chlorine by Bomb Calorimeter - ASTM Method D-808-44/Volume D 05.01 - This method uses a Parr Bomb Calorimeter for the determination of chlorine in lubricating oils and greases, including new and used lubricating oils and greases containing additives, and in additive concentrates. Its range of applicability is 0.1 to 50% chlorine. The procedure assumes that compounds containing halogens other than chlorine will not be present.

Chloride - EPA Method 325.3/USEPA-600/4-79-020 - This is mercuric nitrate titration for the determination of chloride in water and wastewater and saline waters. Iodide and bromide are also measured using this procedure if they are present.

Color - EPA Method 110.3/USEPA-600/4-79-020 - This is a spectrophotometric method that is applicable to all water and wastewater samples, with the exception of very turbid samples. If the sample has turbidity removed by filtration, this test is for true color rather than apparent color.

Conductivity - EPA Method 120.1/USEPA-600/4-79-020 - This is an electrode measurement of current to voltage ratio across a parallel plant cell to determine the conductivity of water and wastewater samples.

Cyanide, Amenable to Chlorination - EPA Method 335.1/USEPA-600/4-79-020 - This method is a manual distillation with magnesium chloride followed by pyridine-barbituric acid colorimetric determination. This procedure is applicable to the determination of cyanide amenable to chlorination in water and wastewater.

Cyanide, Total - EPA Method 335.2/USEPA-600/4-79-020 - This method is a manual distillation with magnesium chloride followed by pyridine-barbituric acid colorimetric determination. This procedure is applicable to the determination of cyanide in water and wastewater. Cyanide is extremely toxic and occurs primarily in industrial effluents. Natural waters do not contain cyanide, and its presence normally indicates contamination from an industrial source. In this procedure, hydrogen cyanide (HCN) is liberated from an acidified sample by distillation and purging with air. The HCN is collected by passing it through a NaOH scrubbing solution. The cyanide concentration from this solution is then determined spectrophotometrically.

Dissolved Oxygen - EPA Method 360.1/USEPA-600/4-79-020 - This is an electrometric method using a membrane electrode. This measurement is based on the rate of diffusion of molecular oxygen across a membrane. This procedure is applicable to water and wastewater samples.

Fecal Coliform by Membrane Filter Techniques - Microbiological Methods for Monitoring the Environment (Water and Waste), USEPA, 1978, Part III, Section C, pp 124-132 - This is a direct membrane filter technique. This procedure is applicable to all water and wastewater samples. Chlorination, disinfection, and toxic materials such as metals, phenols, acids, or caustics affect this test.

Flashpoint - USEPA/SW-846 Method 1010 and ASTM Method D93-77 and ASTM Method D93-80 - This method uses the Pensky-Maartens closed-cup flashpoint tester to determine the flashpoint of fuel oils, lube oils, suspensions of solids, liquids that tend to

form a surface film under test conditions, and other liquids. This sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flashpoint is the lowest temperature at which application of the test flame ignites the vapor above the sample. These test results can be affected by ambient pressure, sample homogeneity, drafts in the area of the tester, and operator bias.

Fluoride - EPA Method 340.1/USEPA-600/4-79-020 - This is a colorimetric procedure for the quantitative determination of fluoride in water and wastewater. The method uses SPADNS reagent for the color development.

Graphite Furnace Elemental Analysis - EPA Methods 202.1, 202.2, 204.1, 204.2, 206.2, 208.1, 208.2, 210.1, 210.2, 213.1, 215.1, 218.1, 218.2, 218.4, 219.1, 219.2, 220.1, 220.2, 231.2, 232.1, 236.1, 236.2, 239.1, 239.2, 242.1, 243.1, 243.2, 245.1, 246.1, 246.2, 249.1, 249.2, 255.1, 255.2, 258.1, 258.2, 270.2, 272.1, 273.1, 279.1, 279.2, 282.1, 282.2, 283.1, 283.2, 286.1, 286.2, 289.1, 289.2/USEPA-600/4-79-020 - This method is an atomic absorption procedure approved for determining the concentration of metals in wastes, mobility procedure extracts, soils, and groundwater. All samples must be subjected to an appropriate dissolution step prior to analysis. Prior to analysis by graphite furnace, samples must be prepared in order to convert organic forms of the elements to inorganic forms, to minimize organic interferences, and to convert the sample to a suitable solution for analysis. The sample preparation procedure varies depending on the sample matrix. Aqueous samples are subjected to an acid digestion procedure. Following the appropriate dissolution of the sample, a representative aliquot of the digestate is spiked with a matrix modifier solution and is placed by means of an autosampler into a graphite tube furnace. The sample aliquot is then slowly evaporated to dryness, charred (ashed), and atomized. the absorption of EDL radiation during atomization will be proportional to the element's concentration.

Hardness - EPA Method 130.2/USEPA-600/4-79-020 - This is a titrimetric method using EDTA. This method is applicable to drinking, surface, and saline waters, as well as domestic and industrial wastes for all concentration ranges of hardness. To avoid large titration volumes, however, a sample aliquot containing not more than 25 mg of Ca CO_3 should be used. Excessive amounts of heavy metals can interfere with the results of this test, but this can be overcome by complexing with cyanide.

Hexavalent Chromium Preparation - USEPA/SW-846 Method 3060 - This is an alkaline digestion preparation for the instrumental determination of hexavalent chromium in solid wastes. This method uses a basic digestion of the waste sample to solubilize both water-insoluble and water-soluble hexavalent chromium compounds. This sample is extracted with hot, 3% sodium carbonate/2% sodium hydroxide solution to dissolve all Cr (VI) and to protect it from reduction to trivalent chromium. Wastes containing high amounts of buffering capability may require additional digestion solution to properly digest the sample.

Hydrazine - Annual Book of ASTM Standards, Part 31, Designation:D1385-78/1981 - This is a colorimetric method for the quantitative determination of hydrazine in water and wastewater. Hydrazine is a man-made chemical and is not found in natural waters. This method is applicable to all water samples but is used mostly on boiler feedwaters, process waters and other waters that have been treated with hydrazine (N_2H_4) for the prevention of corrosion by oxygen. Because of its sensitivity, this method is most often used to determine hydrazine concentrations below 0.1 mg/L and thus requires a dilution of more concentrated samples. While substances normally present in industrial water do not interfere, the hydrazine content may be diminished by the presence of oxidizing agents.

Colors in the prescribed wave lengths also interfere as do other dark colors or turbidities that cannot be overcome by the prescribed treatment.

Mercury - EPA Methods 245.1 and 245.5/USEPA-600/4-79-020 - This method uses flameless, cold vapor, atomic absorption techniques. This procedure measures total mercury in surface and saline water, wastewaters, industrial effluents, sanitary sewage, soils, sediments, bottom deposits, sludges, and EP Toxicity Extracts.

Metals by Atomic Absorption Spectroscopy - EPA Methods 202.1, 202.2, 204.1, 204.2, 206.2, 208.1, 208.2, 210.1, 210.2, 213.1, 215.1, 218.1, 218.2, 218.4, 219.1, 219.2, 220.1, 220.2, 231.2, 232.1, 236.1, 236.2, 239.1, 239.2, 242.1, 243.1, 243.2, 245.1, 246.1, 246.2, 249.1, 249.2, 255.1, 255.2, 258.1, 258.2, 270.2, 272.1, 273.1, 279.1, 279.2, 282.1, 282.2, 283.1, 283.2, 286.1, 286.2, 289.1, 289.2/USEPA-600/4-79-020 - This is an alternate technique (secondary) for the analyses of metals. The primary technique for analysis is inductively coupled plasma spectroscopy. When this method is used, an aqueous sample is aspirated into a nebulizer where the element being determined is reduced to the elemental state, vaporized and imposed in the beam of radiation from a hollow cathode lamp. The AA instruments used in the laboratory are equipped with dedicated data handling systems.

Metals by Inductively Coupled Plasma - EPA Method 200.7/USEPA-600/ 4-79-020 - Inductively Coupled Plasma emission spectroscopy (ICP) determines elements including metals in solution. This is the primary technique for analyses of samples for metals in the laboratory. The method is applicable to a large number of metals and wastes. All matrices, including groundwater, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, and other solid wastes, require digestion prior to analysis. This method describes the sequential, multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by high temperatures in the radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tube. Background correction is used for all trace element determination. Background levels are measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical lines, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured.

Moisture Percentage Determination - EPA Method 160.3/USEPA-600/ 4-79-020 - This is a gravimetric method that determines the total free water in soils and sludges by a weight loss at 100° C. Samples containing volatile solvents or materials which evaporate at 100° C will give a high bias to results.

Nitrogen, Ammonia, by Direct Nesslerization - EPA Method 350.2/USEPA-600/ 4-79-020 - This is a colorimetric determination of ammonia in water and wastewater after color development with Nesslers Reagent.

Nitrogen, Ammonia, Nesslerization after Distillation - EPA Method 350.3/USEPA-600/4-79-020 - This is colorimetric determination of ammonia in water or sludge after distillation using a Kjeldahl Distillation Rack. This color development is done using Nesslers Reagent.

Nitrogen, Nitrate/Nitrite by Rapid Flow Analyzer (RFA) - EPA Methods 353.3 and 354.1/USEPA-600/4-79-020 - This is an automated analysis based on the manual spectrophotometric method for reduction of nitrate to nitrite before color development. Nitrate is quantitatively reduced to nitrite with an open tubular cadmium reactor. The nitrite originally present in the sample plus nitrite reduced from nitrate dazatize with sulfanilamide and coupled with N-(1-naphthyl)-ethylenediamine dihydrochloride to from a red-purple complex measured at 540 nm. In this procedure, nitrogen is used as the segmentation gas. This maintains a constant pH by minimizing the formation of cadmium hydroxide from the reaction of oxygen on the reactor wall. Turbid samples must be filtered prior to analysis. An imidazole buffer is used to eliminate interference due to iron, copper, or other metals.

Odor - EPA Method 140.1/USEPA-600/4-79-020 - This method is applicable to the determination of threshold odor of drinking, surface, and saline waters, domestic and industrial wastes. Due to wide variability of results among analysts and day to day determinations on the same samples, it is recommended that this procedure be conducted by at least five individual observers. Because chlorine may contribute to observers perception of odor, it is desirable to determine odor of a chlorinated sample as well as the same sample after removal of chlorine. Dechlorination is achieved using sodium thiosulfate in exact stoichiometric quantity. It is important to also check a blank to which a similar amount of dechlorinating agent has been added to determine if any odor has been imparted.

Oil and Grease - EPA Method 308.1/USEPA-600/4-79-020 - This is a freon extraction method for the gravimetric determination of oil and grease in water and waste water samples. Freon extractable compounds include waxes, fatty acids, fats, soaps, and other hydrocarbons.

Ortho-Phosphate - EPA Method 365.2/USEPA-600/4-79-020 - This is an ascorbic acid colorimetric determination for ortho-phosphate that is applicable to water and wastewater samples.

Phenols by Rapid Flow Analyzer - EPA Method 420.2/USEPA-600/4-79-020 - This is an automated method for the colorimetric determination of total phenol using the Alpkem Rapid Flow Analyzer (RFA). Phenol distills at 150° C and the distillate reacts with 4-aminoantipyrine and potassium ferricyanide to form a red antipyrine dye. The absorbance is measured at 505 nm. Oxidizing agents, such as chlorine, sulfur compounds, phenol-decomposing bacteria, reducing substances and alkaline pH values interfere with phenol analysis.

Phenols using a Chloroform Extraction - EPA Method 420.1/USEPA-600/4-79-020 - This procedure is for the determination of total phenols in water and wastewater in which the phenol is less than 0.05 mg/L. It is a colorimetric method that uses a preliminary chloroform extraction.

pH Measurement - EPA Method 150.1/USEPA-600/4-79-020 - This is an electrometric determination for pH using a combination electrode. This method is applicable to water and wastewater samples. A coating of oily material or particulate matter can impair the electrode response. If pH must be measured on a solid sample, SW-846 Method 1340 is used for the determination. The method for a solid uses the same electrometric technique using a combination electrode. The pH meter is calibrated daily using 4, 7, and 10 buffer solutions.

Phosphorus, Total - EPA Method 365.2/USEPA-600/4-79-020 - This a colorimetric method for the determination of total phosphorus in water and wastewater samples. The procedure involves a preliminary persulfate digestion.

Reactivities of Hazardous Substances - USEPA/SW-846 Method 9010/30 - This is a method for the determination of reactivities in suspected hazardous substances. The procedure determines: the evolution of cyanide and/or sulfide, reactivity as an acid or base, reactivity as an oxidizer or reducer, reactivity with water, and reactivity with air. Control samples for each classification are analyzed with every batch of unknowns.

Residual Chlorine - EPA Method 330.5/USEPA-600/4-79-020 - This is a colorimetric method for the determination of total residual chlorine in water and wastewater.

Salinity - Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 209 - This is an electrode method for the determination of salinity in water and wastewater. The procedure is based on the determination of the conductivity of the sample. Salinity is an important measurement in the analysis of certain industrial wastes and seawater. It is defined as the total solids in water after all carbonates have been converted to oxides, all bromide and iodide have been replaced by chloride, and all organic matter has been oxidized. It is numerically smaller than the filterable residue and usually is reported as grams per kilogram.

Specific Gravity - Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 210 - Specific gravity is determined by comparing the weight of a volume of the sample of mud, sludge, or industrial waste with the weight of an equal volume of distilled water.

Standard Plate Count - Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 1114 and Microbiological Methods for Monitoring the Environment, Water and Waste, USEPA 1978, Part III, Section A - This is a direct quantitative measurement of bacteria in a water environment. This procedure is applicable to water and wastewater.

Sulfate - EPA Method 375.4/USEPA-600/4-79-020 - This a spectrophotometric method for the determination of sulfate in water and wastewater. Sulfate is widely distributed in nature and may be present in natural waters in concentrations ranging from a few to several thousand milligrams per liter. Mine drainage wastes may contribute large amounts of sulfate through pyrite oxidation. The turbidimetric method is the spectrophotometric method used by the laboratory in this determination. Sulfate ion is precipitated in a hydrochloric acid medium with barium chloride to form barium sulfate crystals of uniform size. Light absorbance of the barium sulfate suspension is measured by a spectrophotometer and the sulfate ion concentration is determined by comparison of the reading with a standard curve.

Surfactants (Methylene Blue Active Substances) - Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 512 - This is a colorimetric method for the determination of surfactants,which is applicable to the measurement of man-made surfactants in drinking waters, surface waters, domestic and industrial wastes over a range of concentrations. Occurrence of materials, other than man-made surface active agents at interference levels is relatively rare and with the exception of chlorides may generally be disregarded. Chlorides at concentrations of about 1000 mg/L show a positive interference and for this reason the method is not applicable to brine samples.

Settleable Solids (SS) - EPA Method 160.5/USEPA-600/4-79-020 - This is a volumetric method using an Imhoff Cone for the determination of settleable solids in water and

wastewater. The method is applicable to surface and saline waters, domestic and industrial wastes.

Total Coliform - Standard Methods for the Examination of Water and Wastewater, 16th edition, 1975, APPHA, AWWA,WPCF, part 1112TMFand Microbiological Methods for Monitoring the Environment, Water, and Waste, USEPA 1978, Part III, Section B - This is a single step membrane filter technique for the determination of total coliform in water and wastewaters.

Total Dissolved Solids (TDS) - EPA Method 160.1/USEPA-600/4-79-020 - This is a gravimetric method for the determination of total dissolved solids in water and wastewater. Total dissolved solids is also referred to as total dissolved residue and filterable residue.

Total Organic Carbon in Water (TOC) - EPA Method 415.1/USEPA-600/4-79-020 - This is an instrumental method for the determination of total organic carbon in water and wastewater samples. This method is based on the UV oxidation of organics in the presence of persulfate.

Total Organic Carbon in Solid Samples (TOC) - EPA Method 415.1/USEPA-600/4-79-020 - This is an instrumental method for the determination of total organic carbon in soil or solid samples using the furnace technique.

Total Solids (TS) - EPA Method 160.3/USEPA-600/4-79-020 - This is a gravimetric procedure for the determination of total solids in water and wastewater samples. Total solids is also referred to as total residue.

Total Volatile Solids (VSS) - EPA Method 160.4/USEPA-600/4-79-020 - This is a gravimetric procedure for the determination of total volatile residue in a water or wastewater sample. The volatile components of a sample can be determined by weighing a dried, filtered sample, igniting the sample to 550° C, cooling the sample, and weighing the sample for a final measurement. The difference in weight represents the total volatile residue of the sample.

Total Suspended Solids (TSS) - EPA Method 160.2/USEPA-600/4-79-020 - This is a gravimetric method for the determination of suspended solids in water and wastewater samples.

Total Kjeldahl and Organically-Bound Nitrogen - EPA Method 351.3 and 351.4/USEPA-600/4-79-020 - This is wet chemistry method for the determination of total kjeldahl nitrogen (TKN) and organically-bound nitrogen in water and wastewater samples. This procedure includes a digestion and distillation followed by Nesslerization. This procedure is the preferred method for the determination of TKN and organically-bound nitrogen in soil, sediments, and sludges. TKN is the sum of free ammonia and organic nitrogen compounds which are converted to ammonium sulfate after digestion. Organic Nitrogen is obtained by the difference of the ammonia nitrogen from the TKN value. The amount of ammonia may be determined before digestion.

Turbidity - EPA Method 180.1/USEPA-600/4-79-020 - This method is for the instrumental determination of turbidity using a turbidimeter. The method is based on the comparison of the intensity of light scattered by the sample with that of a reference standard. The method is applicable to all water and wastewater that is free of debris and coarse sediment.

ORGANIC CHEMISTRY PROCEDURES

Organochlorine Pesticides and PCBs in Water by Gas Chromatography - EPA Method 608/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII] -
This is a gas chromatographic method for the determination of certain organochlorine pesticides and polychlorinated biphenyls in water and wastewater. The analytical method uses a gas chromatograph equipped with an electron capture detector (ECD). Sample preparation requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD.

Organochlorine Pesticides and PCBs in Sediment and Soil by Gas Chromatography - USEPA SW-846 Method 8080 - This is a gas chromatographic method for the determination of certain organochlorine pesticides and polychlorinated biphenyls in water and wastewater. The analytical method uses a gas chromatograph equipped with an electron capture detector (ECD). Sample preparation requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD.

Volatile Halocarbons in Water by Gas Chromatography - EPA Method 601/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a purge and trap gas chromatographic method that is applicable to the determination of halogenated volatile organic compounds. The method is summarized as follows: an inert gas is bubbled through a five milliliter water sample contained in a specially designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is complete, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a Hall electrolytic conductivity detector. The output for these analyses for volatile halocarbons is acquired and analyzed using a chromatography automation package that is part of the laboratory information management system. This computerized option allows data to be reduced automatically after each analysis.

Volatile Aromatics in Water by Gas Chromatography - EPA Method 602/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a purge and trap gas chromatographic method that is applicable to the determination of aromatic volatile organic compounds. The method is summarized as follows: an inert gas is bubbled through a five milliliter water sample contained in a specially designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After purging is complete, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatic volatiles which are then detected with a Photoionization detector. The output for these analyses for volatile halocarbons is acquired and analyzed using a chromatography automation package that is part of the laboratory information management system. This computerized option allows data to be reduced automatically after each analysis.

Volatile Organics in Water by Gas Chromatograph/Mass Spectrometer - EPA Method 624/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) and USEPA SW-846 Methods 8240 - This method covers the determination of a number of volatile organic compounds using a gas chromatograph/mass spectrometer (GC/MS). It uses a purge and trap introduction of the sample. An inert gas is bubbled through a five milliliter water sample contained in a specially designed purging chamber at ambient temperature. The volatile organics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the volatiles are trapped. After purging is complete, the trap is heated and backflushed with the inert gas to desorb the volatiles onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the volatile organics which are then detected with a mass spectrometer detector. The GC/MS system uses a mass spectrometer that is capable of scanning from 20 to 260 amu every seven seconds, utilizing 70 V electron energy in the electron impact ionization mode, and produces a mass spectrum which meets all the tuning criteria specified by the EPA Contract Laboratory Program (CLP) when the instrument is tuned using bromofluorobenzene (BFB). The system is equipped with a computer system that is directly interfaced to the mass spectrometer. This data system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic temperature program. The computer has software that allows searching any GC/MS data file for specific masses and plotting these mass abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. All internal standards and surrogate spikes that are required by the EPA Method 624 and the EPA CLP are used for this analysis.

Base/Neutral and Acid Extractable Compounds in Water and Soil by Gas Chromatograph/Mass Spectrometer - EPA Method 625/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) and USEPA SW-846 Methods 8250 and 8270 - This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. This method includes the GC/MS analysis of chlorinated pesticides, phthalate esters, various neutral compounds, various base compounds, polynuclear aromatic hydrocarbons, phenolic compounds (acid extractables), and polychlorinated biphenyls. Sample preparation requires two liquid/liquid extractions of a one liter sample with methylene chloride. The first extraction is done with a pH adjustment of the sample to greater than 11; the second extraction is done on the same liter of water with a pH adjustment of the sample to less than 2. The individual extracts are filtered into Kuderna Danish Flask/Concentrator tube apparatuses. The extracts are concentrated using Kuderna Danish/Snyder Column Techniques. Sample preparation for solid samples requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. During the instrumental analysis, the gas chromatograph is temperature programmed to separate the base/neutral and acid compounds, which are then detected with a mass spectrometer detector. The GC/MS system uses a mass spectrometer that is capable of scanning from 35 to 450 amu every seven seconds, utilizing 70 V electron energy in the electron impact ionization mode, and produces a mass spectrum which meets all the tuning criteria specified by the EPA Contract Laboratory Program (CLP) when the instrument is tuned using decafluorotriphenyl phosphine (DFTPP). The system is equipped with a computer system that is directly interfaced to the mass spectrometer. This data system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic temperature program. The computer has software that allows searching any GC/MS data file for specific masses and plotting these mass abundances versus time or scan number. This type of plot is defined as an Extracted Ion

Current Profile (EICP). The software is also capable of integrating the abundance in any EICP between specified time or scan number limits. All internal standards and surrogate spikes that are required by the EPA Method 625 and the EPA CLP are used for this analysis.

Chlorinated Phenoxy Acid Herbicides in Water - Standard Methods for the Analysis of Water and Wastewater, 15th Edition, Method 509-A - This is a gas chromatographic method for quantitatively determining certain chlorophenoxy acid herbicides in water. The instrumental analysis uses a gas chromatograph equipped with an electron capture detector (ECD). Chlorinated phenoxy acids and their esters are extracted from an acidified one liter sample of water with diethyl ether. The esters are hydrolyzed to acids and extraneous organic material is removed by a solvent wash. The acids are converted to methyl esters which are extracted from the aqueous phase. The extract is cleaned up by passing it through a micro-adsorption phase. Identification of the esters is made by selective gas chromatographic separations and is corroborated through the use of two or more unlike columns. Detection and measurement is accomplished by ECD. Since these compounds occur in water in various form, i.e. acid, salt, ester, etc., a hydrolysis step is included to permit the determination of the active part of the herbicide.

Chlorinated Phenoxy Acid Herbicides in Soil - Standard Methods for the Analysis of Water and Wastewater, 15th Edition, Method 509-A -Adaptation - This is a gas chromatographic method for quantitatively.determining certain chlorophenoxy acid herbicides in water. The instrumental analysis uses a gas chromatograph equipped with an electron capture detector (ECD). Chlorinated phenoxy acids and their esters are extracted from the acidified sample with diethyl ether. The esters are hydrolyzed to acids and extraneous organic material is removed by a solvent wash. The acids are converted to methyl esters which are extracted from the aqueous phase. The extract is cleaned up by passing it through a micro-adsorption phase. Identification of the esters is made by selective gas chromatographic separations and is corroborated through the use of two or more unlike columns. Detection and measurement is accomplished by ECD. Since these compounds occur in water in various form, i.e. acid, salt, ester, etc., a hydrolysis step is included to permit the determination of the active part of the herbicide.

Phenols in Water and Solids by Gas Chromatography - EPA Method 604/USEPA-600/4-79-020 (40 CFR, Part 136 [Part VIII] October 26,1984) and USEPA SW-846 Methods 8040 - This is a gas chromatographic method for the determination of phenols (acid extractables) in liquid and solid matrices. The instrumental analysis uses a gas chromatograph equipped with a flame ionization detector and a special column that is deactivated for acid compounds. If this deactivated column in not used, the phenolic compounds tend to produce a tailing effect that makes the quantitations difficult and less accurate than when this precaution is enforced. Sample preparation for liquid samples requires a liquid/liquid extraction of a one liter sample with a pH adjustment to less than 2. The extract is filtered in a Kuderna-Danish/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/FID.

Polychlorinated Biphenyls in Transformer Oil - EPA Method entitled Analysis of Transformer Oils for Polychlorinated Biphenyls - This is a gas chromatographic method for quantitatively determining polychlorinated biphenyls in transformer oils and other oils. The instrumental analysis for this procedure requires a gas chromatograph equipped with an electron capture detector (ECD). Sample preparation for this method requires a dilution of the pure oil, followed by a micro-alumina cleanup, before instrumental analysis. Second column confirmations must be made on samples that are believed to contain PCBs from the initial run.

Organophosphorus Pesticides by Gas Chromatography - Standard Methods for the Analysis of Water and Wastewater, 15th Edition, Method 509-A. This is a gas chromatographic method for the determination of certain organophosphorus pesticides in water and wastewater. The analytical method uses a gas chromatograph equipped with an electron capture detector (ECD). Sample preparation requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. Sample preparation for a solid sample requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. This extract may also be analyzed by GC/MS using the same technique as in the analysis of base/neutral and acid extractable organics.

Industrial Solvents by Flame Ionization Detector - USEPA SW-846 Method 8010, 8020, and 8240 - This is a gas chromatographic method for the quantitative determination of certain solvents in waste materials and solids. This method is generally used for high level solvents within the matrix and is therefore done using a flame ionization detector on a gas chromatograph. Since the method is applicable to high levels, the purge and trap sample introduction technique is not used. A liquid/liquid extraction or a solid/liquid extraction is used to prepare the sample for direct solvent injection into the gas chromatograph.

Hydrocarbon Analysis by Flame Ionization Detector - Annual Book of ASTM Standards, Petroleum Products and Lubricants, Volume 05.01, pp 326-330, 1983 - This is a gas chromatographic method using a flame ionization detector for the quantitative determination of hydrocarbons in various matrices. The various hydrocarbons that are included in this method are kerosene, gasoline, diesel fuel (No. 2 fuel oil), No. 5 fuel oil, JP-4, JP-5, mineral spirits, odorless mineral spirits, and naphtha. Samples are extracted into carbon disulfide and this extract is directly injected into the GC/FID. This procedure may be used either to fingerprint a substance against a known fingerprint or to quantitate based on a known standard.

Chlorinated Hydrocarbons by GC/ECD - EPA Method 612/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is gas chromatographic method for the determination of certain chlorinated hydrocarbons using a gas chromatograph equipped with an electron capture detector (GC/ECD). Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables.

Phthalate Esters in Water and Solids - EPA Method 606/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic method for the quantitative determination of certain phthalate esters in water and solids using a gas chromatograph equipped with an electron capture detector. Sample preparation for liquids requires a

liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables.

Haloether by Gas Chromatography - EPA Method 611/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic method for the quantitative determination of certain haloethers in municipal and industrial discharges using a gas chromatograph equipped with an electron capture detector (GC/ECD). Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables.

Organic Vapor Monitoring by Gas Chromatograph - Organic Vapor Monitor Manual, 3M™ Company, used with the 3M™ #3500 badges before analysis - This is a gas chromatographic method using a flame ionization detector for the determination of organic vapors from the 3M™ Organic Vapor Monitors. The 3M™ Organic Vapor Monitor is a charcoal wafer that desorbs from the atmosphere which the wearer is exposed to. The organic vapor constituents can be extracted from the badge using carbon disulfide. The carbon disulfide extract is analyzed for a number of organic solvents using GC/FID.

Acrolein, Acrylonitrile, and Acetonitrile - EPA Method 603/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic method using a flame ionization detector for the quantitative determination of acrolein, acrylonitrile, and acetonitrile in municipal and industrial discharges. The sample is extracted with carbon disulfide and directly injected in the GC/FID. An alternative to this method is analysis of the sample by GC/MS using the same techniques as for volatile organics as outlined in the EPA Method 624.

Nitroaromatics and Isophorone by Gas Chromatography - EPA Method 609/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is gas chromatographic method for the quantitative determination of certain nitroaromatics and isophorone using gas chromatography. A flame ionization detector is used to determine isophorone and nitrobenzene. An electron capture detector is used to determine the dinitrotoluenes. Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD or GC/FID. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna

Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/ECD or GC.FID. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables. This GC/MS procedure is outlined in EPA Method 625.

Nitrosamines by Gas Chromatography - EPA Method 607/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic method for the quantitative determination of certain nitrosamines using a gas chromatograph equipped with a Hall electrolytic conductivity detector in the nitrogen mode. Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/Hall in Nitrogen mode. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/Hall in Nitrogen mode. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables. This GC/MS procedure is outlined in EPA Method 625.

2,3,7,8-Tetrachlorodibenzo-p-Dioxin - EPA Method 613/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic/mass spectrometer method for the determination of 2,3,7,8-tetrachlorodibenzo-p-dioxin in municipal and industrial discharges.Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/MS. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/MS.

Anesthesia Gases in Air Samples - NIOSH Method - This is a gas chromatographic method for the quantitative determination of certain anesthesia gases in air samples. The collection of anesthesia gases is performed onto activated charcoal in glass collection tubes using a sampling vacuum pump. This activated charcoal is extracted with carbon disulfide and the extract is analyzed by gas chromatography using a flame ionization detector.

Creosote by Gas Chromatography - EPA Method 610/USEPA-600/4-79-020 (40 CFR, Part 136 Part VIII) - This is a gas chromatographic method for the quantitative determination of creosote in oil, sludge, and water. Creosote is an ambiguous name for two specific mixtures. Wood creosote is a mixture of phenols obtained from wood tar. This type of creosote can be extracted and analyzed by the gas chromatographic phenol method. Creosote from coal tar is called coal tar creosote. Coal tar creosote is a distillate of coal tar produced by high temperature carbonization of bituminous coal. the constituents of coal tar creosote are liquid and solid polynuclear aromatic hydrocarbons, tar acids (up to 3%), and tar bases. This type of creosote is a translucent brown to black, oily liquid. It has a characteristic sharp odor. It is heavier than water. Creosote has a flash point of 165° F and an ignition temperature of 637° F. Creosote is practically insoluble in water. Creosote is used for impregnating wood to protect it from rot and worms; it is also used as a disinfectant, insecticide, fungicide, and germicide. Sample preparation for liquids requires a liquid/liquid extraction of one liter of the sample with methylene chloride. The

extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/FID in Nitrogen mode. Sample preparation for solids requires a solid/liquid extraction using an ultrasonic extractor. The extract is filtered into a Kuderna Danish Flask/Concentrator tube apparatus. The extract is concentrated using Kuderna Danish/Snyder Column Techniques. After a final nitrogen blowdown, the extract is reconstituted with iso-octane for analysis by GC/FID. The alternate method of analysis for this class of compounds is analysis by GC/MS as described in the analysis of base/neutral and acid extractables. This GC/MS procedure is outlined in EPA Method 625.

Table 1
Detection Limit Values - Metals

Parameter	Detection Limits
Aluminium	0.05 ppm
Antimony	0.01 ppm
Arsenic	0.005 ppm
Barium	0.1 ppm
Beryllium	0.1 ppm
Boron	0.05 ppm
Cadmium	0.01 ppm
Calcium	0.1 ppm
Chromium	0.5 ppm
Cobalt	0.1 ppm
Iron	0.05 ppm
Lead	0.02 ppm
Lithium	0.1 ppm
Magnesium	0.1 ppm
Manganese	0.05 ppm
Mercury	0.0002 ppm
Molybdenum	0.1 ppm
Neodymium	0.1 ppm
Nickel	0.05 ppm
Niobium	0.1 ppm
Palladium	0.1 ppm
Phosphorous	0.5 ppm
Potassium	0.5 ppm
Selenium	0.005 ppm
Silicon	0.5 ppm
Silver	0.05 ppm
Sodium	0.1 ppm
Strontium	0.05 ppm
Sulfur	0.1 ppm
Thallium	0.02 ppm
Thorium	0.05 ppm
Tin	0.1 ppm
Titanium	0.1 ppm
Uranium	0.1 ppm
Vanadium	0.1 ppm
Zinc	0.05 ppm

Table 2
Detection Limit Values - Wet Chemistry

Parameter	Detection Limits
Acidity	NA
Alkalinity	NA
BOD	1 ppm
Chloride	1 ppm
Chlorine	0.1 ppm
COD	10 ppm
Conductance (Specific)	NA
Corrosivity	NA
Cyanide	0.1 ppm
Flash Point	>140° F
Fluoride	1.0 ppm
Formaldehyde	0.1 ppm
Hardness (Total)	0.1 ppm
Heating Value	NA
Nitrogen (Ammonia)	1 ppm
Nitrogen (Kjeldahl)	1 ppm
Nitrogen (Nitrate)	1 ppm
Nitrogen (Nitrite)	1 ppm
Odor	NA
Oil&Grease	1 ppm
pH	NA
Phenol	20 ppb
Phosphorous (Ortho)	1 ppm
Phosphorous (Total)	1 ppm
Reactivity	NA
Solids (Dissolved)	1 ppm
Solids (Setteable)	1 ppm
Solids (Suspended)	1 ppm
Solids (Total)	1 ppm
Solids (Volatile)	1 ppm
Sulfate	1 ppm
Sulfide (H_2S)	0.1 ppm
Sulfite	2.0 ppm
Surfactants (MBAS)	0.1 ppm
Turbidity	NA
TOC	1.0 ppm
TOX	0.01 ppm

Table 3
Detection Limit Values - Organics

Parameter	Detection Limits
Acenaphthene	10 ppb
Acenaphthylene	10 ppb
Acetone	10 ppb
Acrolein	10 ppb
Acrylonitrile	10 ppb
Anthracene	10 ppb
Benzene	10 ppb
Benzidine	10 ppb
Benzo(a)anthracene	10 ppb
Benzoic Acid	10 ppb
Benzo(a)pyrene	10 ppb
Benzo(b)fluoranthene	10 ppb
Benzo(ghi)perylene	10 ppb
Benzo(k)fluoranthene	10 ppb
Benzyl Chloride	10 ppb
Benzyl Butyl Phthalate	10 ppb
Biphenyl	10 ppb
Bis(2-chloroethoxy) methane	10 ppb
Bis(2-chloroethyl) ether	10 ppb
Bis(2-ethylhexyl)phthalate	10 ppb
Bromodichloromethane	10 ppb
Bromomethane	10 ppb
Bromoform	10 ppb
4-Bromophenylphenyl ether	10 ppb
Carbazole	10 ppb
Carbon tetrachloride	10 ppb
Chloroethane	10 ppb
4-Chloro-3-methylphenol	10 ppb
Chlorobenzene	10 ppb
2-Chloroethylvinyl ether	10 ppb
Chloroform	10 ppb
Chloromethane	10 ppb
2-Chloronaphthalene	10 ppb
2-Chlorophenol	10 ppb
4-Chlorophenylphenyl ether	10 ppb
Chrysene	10 ppb
p-Cymene	10 ppb
n-Decane	10 ppb
Dibenzo(a,h)anthracene	10 ppb
Dibenzofuran	10 ppb
Dibenzothiophene	10 ppb
Dibromochloromethane	10 ppb
1,2-Dichlorobenzene	10 ppb
1,3-Dichlorobenzene	10 ppb
1,4-Dichlorobenzene	10 ppb
3,3'-Dichlorobenzidine	10 ppb
Dichlorodifluoromethane	10 ppb
1,1-Dichloroethane	10 ppb
1,2-Dichloroethane	10 ppb
1,1-Dichloroethene	10 ppb
trans-1,2-Dichloroethene	10 ppb

Table 3
Detection Limit Values - Organics

2,4-Dichlorophenol	10 ppb
1,2-Dichloropropane	10 ppb
cis-1,3-Dichloropropene	10 ppb
trans-1,3-Dichloropropene	10 ppb
Diethyl ether	10 ppb
Diethyl phthalate	10 ppb
2,4-Dimethylphenol	10 ppb
Dimethyl phthalate	10 ppb
Di-n-butly phthalate	10 ppb
Di-n-octyl phthalate	10 ppb
2,4-Dinitrophenol	10 ppb
2,4-Dinitrotoluene	10 ppb
2,6-Dinitrotoluene	10 ppb
p-Dioxane	10 ppb
Diphenylamine	10 ppb
Diphenyl ether	10 ppb
1,2-Diphenylhydrazine	10 ppb
n-Docosane	10 ppb
n-Dodecane	10 ppb
n-Eicosane	10 ppb
Epichlorohydrin	10 ppb
Ethylbenzene	10 ppb
Fluoranthene	10 ppb
Fluorene	10 ppb
Hexachlorobenzene	10 ppb
Hexachlorobutadiene	10 ppb
Hexachlorocyclopentadiene	10 ppb
Hexachloroethane	10 ppb
n-Hexacosane	10 ppb
n-Hexadecane	10 ppb
Hexanoic acid	10 ppb
Ideno(1,2,3-cd)pyrene	10 ppb
Isophorone	10 ppb
Methylene Chloride	10 ppb
2-Methyl-4,6-Dinitrophenol	10 ppb
Methyl ethyl ketone	10 ppb
Naphthalene	10 ppb
ß-Naphthylamine	10 ppb
Nitrobenzene	10 ppb
2-Nitrophenol	10 ppb
4-Nitrophenol	10 ppb
N-Nitrosodimethylamine	10 ppb
N-Nitosodi-n-propylamine	10 ppb
N-Nitrosodiphenylamine	10 ppb
n-Octacosane	10 ppb
n-Octadecane	10 ppb
2,2-oxybis(1-chloropropane)	10 ppb
PCB-1016	10 ppb
PCB-1221	10 ppb
PCB-1232	10 ppb
PCB-1242	10 ppb
PCB-1248	10 ppb
PCB-1254	10 ppb

Table 3
Detection Limit Values - Organics

PCB-1260	10 ppb
Pentachlorophenol	10 ppb
Phenanthrene	10 ppb
Phenol	10 ppb
α-Picoline	10 ppb
Pyrene	10 ppb
Styrene	10 ppb
α-Terpineol	10 ppb
2,3,7,8-TCDD	10 ppb
1,1,2,2-Tetrachloroethane	10 ppb
Tetrachloroethene	10 ppb
n-tetracosane	10 ppb
n-Tetradecane	10 ppb
Toluene	10 ppb
n-Triacontane	10 ppb
1,2,3-Trichlorobenzene	10 ppb
1,2,4-Trichlorobenzene	10 ppb
1,1,1-Trichloroethane	10 ppb
1,1,2-Trichloroethane	10 ppb
Trichloroethene	10 ppb
Trichlorofluoromethane	10 ppb
2,3,6-Trichlorophenol	10 ppb
2,4,5-Trichlorophenol	10 ppb
2,4,6-Trichlorophenol	10 ppb
Vinyl Chloride	10 ppb

Table 4
Detection Limit Values - Pesticides

Parameter	Detection Limits
Aldrin	0.0002 ppm
α-BHC	0.0005 ppm
β-BHC	0.0005 ppm
δ-BHC	0.0005 ppm
Lindane (γ-BHC)	0.0004 ppm
Chlordane	0.005 ppm
DDD	0.0005 ppm
DDE	0.0005 ppm
DDT	0.0005 ppm
Dieldrin	0.0005 ppm
Endosulfan I	0.0005 ppm
Endosulfan II	0.0005 ppm
Endosulfan Sulfate	0.0005 ppm
Endrin Aldehyde	0.0005 ppm
Heptachlor	0.0005 ppm
Heptachlor Epoxide	0.0005 ppm
Toxaphene	0.005 ppm

SECTION VIII

DATA REDUCTION, VALIDATION, AND REPORTING

VIII. DATA REDUCTION, VALIDATION, AND REPORTING

Analytical data is generated from the various instruments and analytical procedures. Data in the raw form varies from procedure to procedure. Raw analytical data for organic analyses may be in the form of a chromatogram or a mass spectrum from a complex GC/MS system. Raw analytical data for metals analyses may be absorption or emission data for a particular wavelength. Raw analytical data for wet chemistry analyses may be response of a color-developed standard or sample using spectrophotometric techniques. The data from any of these classes of analyses must be taken from its raw form and translated to a usable form that can be understood by the client and other laymen who might have to use the data. The interpretation of raw data and the calculations associated with transforming raw data into usable data is known as data reduction.

Since data reduction involves the manipulation of many numbers and calculations, it is a prime source for the occurrence of errors. The use of integrated data systems with the measurement instrumentation is one of the best ways to avoid mathematical errors that occur often during the manual manipulation of numbers in the calculations of results.

The automatic data systems and the Laboratory Infomation Management System used by General Engineering Laboratories are described in Section V. Figure VIII-1 shows the flow of the sample through the laboratory, including the data review process after the data has been reduced.

Data validation is the process whereby it is established that the performance of the method at that point in time meets the quality standards established. It is a process which must be planned before the analysis and carried out concurrently with the analysis. For non-routine or unusual samples however, the appropriate data or results validation may have to be decided at the time of analysis. The approaches to data validation are discussed in Section IX.

Validation of Metals Data

For metals analysis a calibration curve is first established for each element to be analyzed within a concentration range generally differing no more than approximately two and one half orders of magnitude from the lowest to highest concentration point of the curve. For samples outside this range a new calibration is required. Once calibrated for the expected concentration range of the element of interest, samples are run and data validated by use of a spiked sample and duplicate for each ten analyses. Samples are spiked to at least fifty percent of the anticipated concentration of the element being analyzed and percent recovery of spike determined as outlined in the following section.

Calculation of reported values involves only the adjustment of instrument readout concentrations to compensate for any sample dilutions and standard recovery factors:

$$C_{RPT} = R \times (F_1/I_1) \times (F_2/I_2) \times (C_{STO}/C_{OBS}) \times f_u$$

C_{RPT} = Concentration reported in mg/L

R = Instrument readout in mg/L

F_1 = Final volume in mL of sample after digestion

I_1 = Initial volume in mL of liquid samples or Initial weight in grams of solid samples

F_2 = Final volume in mL of sample aliquot after dilution

I_2 = Initial volume in mL of sample aliquot

C_{STO} = Known concentration of standard in mg/L

C_{OBS} = Observed concentration of standard in mg/L

f_u = Cancelling factor required to maintain reporting concentration units (C_{RPT}) of mg/L when I_1 units are in grams

Validation of Organic Chemistry Data

For Organics and pesticide analyses data evaluation includes the analysis of laboratory replicates to validate the precision of the analysis and fortified or spiked samples to validate the accuracy of the analysis. For methods 601-612 this consists of mandatory spiking of 10% of all samples and comparing the degree and precision of spike recovery with the test procedure criteria established in the multiple-laboratory methods studies (See section on QA objectives). For Methods 624 and 625 the requirement is for a 5% mandatory spiking of samples.

A generalized formula for the calculation of results for procedures 601-612 is:

$$C_{RPT} = [(V_{STD})(V_F)(P_{SD})(C_{STD})]/[(V_{SA})(V_I)(P_{STD})] \times DF$$

Where:

C_{RPT} = Concentration reported in µg/L

V_{SA} = Volume of sample injected in µL

V_{STD} = Volume of standard injected in µL

V_F = Final volume of sample extract in mL

V_I = Initial volume of sample in mL

P_{SA} = Peak height of sample in nm

P_{STD} = Peak height of standard in nm

C_{STD} = Concentration standard of µg/L

DF = Dilution Factor

For Methods 624 and 625 the calculation for reporting results in µg/L is:

$$C_{RPT} = [(A_S)(C_{IS})(V_{SA})(V_F)]/[(A_{IS})RF(V_{STO})(V_I)] \times DF$$

Where:

C_{RPT} = Concentration reported in µg/L

A_S = Area of the characteristic m/z for the parameter to be measured

A_{IS} = Area of the characteristic m/z for the internal standard

C_{IS} = Concentration of internal standard in µg/L

V_{SA} = Volume of sample injected in µL

V_{STO} = Volume of standard injected in µL

V_F = Final volume of sample in mL

V_I = Initial volume of sample in mL

DF = Dilution Factor

RF = Reference factor calculated from standard using

$$RF = [(A_S)(C_{IS})]/[(A_{IS})(C_S)]$$

Where:

C_S = Concentration of parameter to be measured

For each wet chemistry method which lends itself to evaluation by recovery studies at least one control sample, spiked preferably to twice the anticipated concentration level of the parameter of interest, is run with each batch of ten samples together with a duplicate and a blank.

For colorimetric methods results are calculated by directly comparing peak heights of samples with those of a standard calibration curve of peak heights of standards versus concentration.

The generalized relationship is:

$$\text{mg/L from Standard curve} \times f_D = \text{mg/L reported}$$

Where f_D = any dilution factor that must be taken into consideration in determining the original concentration of a diluted sample.

New standard curves are established with each new batch of reagents, using at least FIVE concentration levels to insure proper curve fit to the data, and for each batch of subsequent sample analyses, one midpoint standard is determined for continued evaluation of the curve and procedure.

For titration procedures a generalized formula for the calculation of results is:

$$C = \frac{A-B}{S} \times N$$

Where:

C = Concentration of parameter of interest in mg/L

A = mL of titrant for sample

B = mL of titrant for blank

N = Normality of titrant

Data Reporting Considerations

In the review and reduction of analytical data before releasing and reporting of the data, significant figures and rounding off of the raw sample results is an important consideration.

Significant Figures

Significant figures should not be reported beyond the accuracy of the specific procedure. Reported analytical values should contain only significant figures. Values reported with more significant figures than warranted by the procedure are misleading and reflect adversely on the credibility of the laboratory.

A value is made up of significant figures when it contains all digits known to be true and one last digit which is in doubt. For example, if a value if reported as 18.8, the 18 is known to be true and the 0.8 is uncertain but thought to be more correct than 0.7 or 0.9.

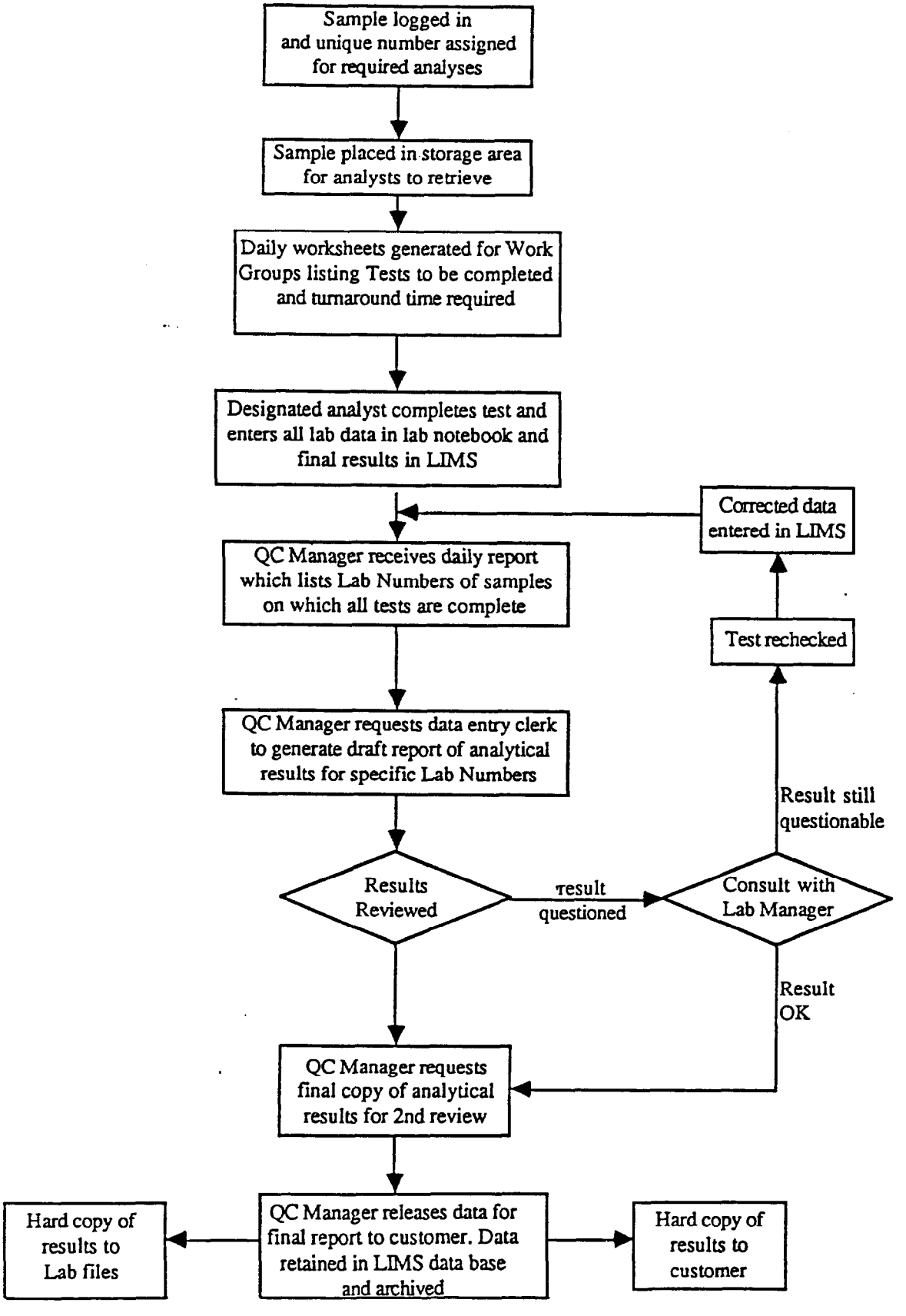
Rounding Off

Since all laboratory calculations are done either directly in the computer or on a calculator, the analyst must generally round off the final answer to the appropriate number of significant figures. Specifically, the number of significant figures depends on the sensitivity of the test. Generally, the following guidelines apply to rounding off:

- Rounding off should be done only on the figure to be reported. Extra figures should be carried through the calculation in order to prevent the loss of meaningful accuracy.
- If the figure following those to be retained is less than 5, it is dropped and the retained figures are left unchanged. (e.g. 11.444 becomes 11.44)
- If the figure following those to be retained is greater than 5, it is dropped and the last retained figure is increased by 1. (e.g. 11.446 becomes 11.45)
- If the figure following those to be retained is equal to 5, it is dropped and the last retained figure is left unchanged if it is even, or it is increased by one if it is odd. (e.g. 11.445 becomes 11.44, but 11.455 becomes 11.46)

Figure VIII-1

LABORATORY INFORMATION MANAGEMENT SYSTEM (LIMS) FOR SAMPLE TRACKING AND DATA FLOW



SECTION IX

LABORATORY QUALITY CONTROL CHECKS

IX. LABORATORY QUALITY CONTROL CHECKS

Overview

Because of an increased concern for hazardous chemicals in the environment and the effects of these chemicals on plant, animal, and human life, there has been a growing recognition of the need to (1) improve the ability to determine data quality for environmentally-related analyses and (2) improve the means of defining data quality related to environmental analysis.

The objectives of the General Engineering Quality Assurance (QA) effort are to:

1. Define the quality of data produced through monitoring the precision and accuracy of the analytical results.
2. Improve the quality of data.
3. Monitor technical compliance with contractual QA requirements.
4. Evaluate laboratory performance.
5. Evaluate method performance and to identify weak methodologies and provide a continuing source of correctives action aimed at overcoming deficiencies..
6. To detect training needs within the analytical group.
7. To provide a permanent record of instrument performance as a basis for validating data and projecting repair or replacement needs.
8. To upgrade the overall quality of laboratory performance.

The Data Base for QA/QC Checks

The QA/QC Data Base is the focal point for QA/QC data storage. The Data Base actually consists of three separate data bases:

1. Metals Analytical Data
2. General Inorganics Analytical Data
3. Organics Analytical Data
   - A. Semi-volatile Extractable Data
   - B. Volatile Data
   - C. Pesticide Data

From the Data Base, internal laboratory control and relative laboratory performance can be evaluated. Performance trends and defects can then be monitored within our laboratory or between our laboratory and any other commercial or regulatory laboratory that is operating under the same analytical protocols with the same Quality Control requirements. Performance-based QC criteria (e.g., acceptance windows for spike recoveries and surrogate spike recoveries) can be evaluated and updated as needed.

The Data Base has provided the information needed to establish performance-based criteria. A full service environmental testing facility, such as General Engineering Laboratories, produces an enormous amount of analytical data from a large number of industries and other clients. This vast empirical data set is carefully analyzed, with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of quality control and performance criteria specifications of what is routinely achievable and expected of state of the art analytical chemistry laboratories in mass production analysis of acutely sensitive environmental samples.

Metals Analyses Quality Control Checks

The EPA 600 Series Protocols and the SW-846 Methodologies outline the criteria for the QA/QC actions required for metals analyses in environmental samples. The following Quality Control Checks are provided by our laboratory for analysis of metals:

1. Initial and continuing calibration.
2. Method blanks.
3. ICP interference check sample.
4. Matrix/digestion spike recovery.
5. Duplicates.
6. Instrument detection limits/laboratory control sample.
7. Standard addition results where applicable.
8. Periodic analysis of an EPA Reference Sample.

General Inorganic Analyses Quality Control Checks

The EPA 600 Series Protocols and the SW-846 Methodologies outline the criteria for the QA/QC actions required for general inorganic analyses in environmental samples. The following Quality Control Checks are provided by our laboratory for analysis of general inorganic chemistry parameters:

1. Initial and continuing calibration.
2. Method blanks.
3. Matrix spike recovery.
4. Blank spike recovery.
5. Duplicates.
6. Established detection limits/laboratory control sample.
7. Periodic analysis of an EPA Reference Sample.

Organic Analyses Quality Control Checks

The criteria for the QA/QC actions required for analysis of environmental samples for organic parameters are explicitly outlined in the EPA 600 Series Methodologies and the EPA SW-846 Methodologies. The following Quality Control Checks are provided by our laboratory for analyses of samples for organic chemistry parameters:

1. Percent recovery for surrogate spikes.
2. Percent recovery for blank spikes.
3. Matrix spike and duplicate recovery.
4. Method blanks.
5. GC/MS tuning and mass calibration, BFB and DFTPP.
6. Initial calibration data and response factor documentation.
7. Continuing calibration check data (response factor).
8. Periodic analysis of EPA Reference sample.

QUALITY ASSURANCE/QUALITY CONTROL FOR ANALYSIS OF METALS

The main objective of the rigorous QA/QC requirements incorporated in the EPA Protocols is to assure the production of data of known quality which will meet the client's needs whether it be to support a remedial decision or an enforcement action. The high level of documentation required in conjunction with the built-in QA/QC serves to provide the laboratory or the regulatory agency with legally defensible data. The QA/QC aspects

contained within the EPA Methodologies may be divided into two categories: (1) instrument or system performance QA/QC and (2) method performance QA/QC.

Instrument Performance QA/QC for Analysis of Metals

- Instrument Calibration - The objective in establishing compliance requirements for satisfactory instrument calibration is to insure that the instrument is capable of producing acceptable quantitative data. The EPA Methodologies specify that instruments must be calibrated daily and each time the instrument is set up. The number of calibration standards are specified for each analytical method. Calibration standards must be prepared using the same type of acid or combination of acids and at the same concentration as will result in the samples following preparation. In addition, for AA analyses, the laboratory is required to analyze a standard at the required detection limit to verify their capability to meet the required levels of detection.

- Initial Calibration Verification - Initial calibration verification (ICV) is performed immediately following instrument calibration by analyzing an independent, certified solution to verify and document the accuracy of the initial calibration for each analyte. When ICV results exceed the specified control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The Initial and Continuing Calibration Control Limits for Metals Analyses are shown in Table 1 below:

Table 1 - Initial and Continuing Calibration Control Limits for Metals Analysis

Analytical Method	Inorganic Species	% of True Value LOW	% of True Value HIGH
ICP/AA	Metals	90	110
Cold Vapor	Mercury	80	120

- Calibration Blank - A calibration blank (CB) is analyzed each time the instrument is calibrated, at the beginning and end of the run, and at a frequency of 10% during the run. The CB serves as a monitor of instrument drift and possible memory effects of contamination. If the CB result is greater than the required detection limit, the method requires that the analysis be terminated, the problem identified and corrected, and the instrument recalibrated.

- Continuing Calibration Verification - To assure calibration accuracy and monitor instrument performance during each analysis run, a continuing calibration verification (CCV) standard is analyzed at a frequency of 10% or every two hours, whichever is more frequent. The analyte concentration in the CCV standard must be at or near the mid-range levels of the calibration curve. When CCV results exceed the specified control limits, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified.

- ICP Interference Check Sample Analysis - The ICP Interference Check Sample (ICS) analysis is performed to verify the ICP's interelement and background correction factors. An ICS, preferably supplied from EPA, must be analyzed at the beginning and end of each analysis run or at a minimum of twice per eight-hour working shift, whichever is more frequent. If the results of the ICS do not

meet the specified control limit of ±20% of the true value, the analysis must be terminated, the problem corrected, the instrument recalibrated, and the samples reanalyzed.

- ICP Linear Range Analysis - To verify linearity near the required detection limit, the laboratory must analyze an ICP standard at a concentration level of two times the required detection limit. This standard must be run at the beginning and end of each sample analysis run or at a minimum of twice per eight-hour working shift, whichever is more frequent. The upper limit of the ICP linear range must be verified on a quarterly basis. The analytically determined concentration of the standard used to define this upper limit must fall within ±5% of the true value. This concentration defines the limit beyond which results cannot be reported without dilution of the sample into the working range.

- Instrument Detection Limits - Instrument detection limits (IDL) must be determined by multiplying by three, the average of the standard deviations obtained on three nonconsecutive days from the analysis of a standard solution (each analyte in reagent water) at a concentration 3-5 times the IDL, with seven consecutive measurements per day. Updated IDLs are documented on a quarterly basis.

Method Performance QA/QC for Analysis of Metals

- Preparation Blanks - The analysis of preparation blanks provides the laboratory and the regulatory agencies a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. At least one preparation blank, consisting of deionized water processed through each sample preparation procedure performed, must be prepared and analyzed with each batch of samples digested or for each 10 samples received. If the concentration of the blank is above the required detected limit, then any samples with less than 10 times the concentration level identified in the blank must be redigested and reanalyzed. The reported sample results are not corrected for the blank results.

- ICP Serial Dilution Analysis - Serial dilution analysis is performed to ascertain whether significant physical or chemical interferences exist due to the sample matrix. One sample from each group of samples of a similar matrix type must undergo at least one serial dilution analysis. If the analyte concentration is minimally a factor of 10 above the IDL after dilution, the analysis must agree within 10% of the original determination. If the result is not within 10%, a chemical or physical interference effect should be suspected and the data flagged.

- Laboratory Control Sample Analysis - The laboratory control sample (LCS) analysis is designed to serve as a monitor of the efficiency of the digestion procedure. An aqueous LCS must be anlayzed on a digestion batch basis, whereas a solid LCS is analyzed on a monthly basis. Results of the LCS analyses must fall within the EPA established control limits. Results which fall outside the specified control limits are indicative of an analytical problem related to the digestion/sample preparation procedures and/or instrument operation. In the EPA Contract Laboratory Program (CLP), the Laboratory Control Samples are provided as needed.

- Spiked Sample Analysis - Spiked sample analysis is designed to provide information about the effect of the sample matrix on the degestion and measurement methodology. The spike is added prior to digestion steps. Spiked

sample analyses are performed for each matrix and concentration level classification for each batch of ten samples. Analyte spiking levels are specified in the EPA Methodologies and the EPA Contract Laboratory specifications. A variety of factors can impact the outcome of the spike sample results; these include matrix suppression or enhancement effects, duplicate precision, digestion efficiency, contamination and the relative levels of analyte in the sample and the spike. If the spike recovery falls outside the limits of 75-125%, the data associated with that spiked sample must be flagged.

- Duplicate Sample Analysis - Duplicate sample analysis is performed for each matrix and concentration level classification within a batch of samples. The results of the duplicate anlayses serve as an indicator of the precision of the method and the sample results. The non-homogeneous nature of typical hazardous waste related samples has made it impractical to define required limits in the EPA Methdologies or the EPA CLP Specifications. As it is difficult to determine whether poor precision is a result of sample non-homogeneity, method inadequacies or laboratory technique, the control limits given in the EPA Protocols are to be used for flagging data rather than for taking corrective action.

- Furnace Atomic Absorption QC Analysis - Duplicate injections and post digestion analytical spikes are incorporated into the QC scheme in order to establish a mechanism whereby the data user can estimate the precision and accuracy of the individual analytical determination relative to the overall method precision and accuracy.

The use of standardized analytical methodology which incorporates rigorous QA/QC and documentation requirements, has served to consistently provide laboratories and regulatory agencies with data of known quality. Our laboratory, the EPA, and other regulatory agencies are constantly striving to improve the quality of the data produced, by continually reviewing, updating, and refining the current methods to accommodate advanced technology and by implementing new methods based upon environmental needs.

QUALITY ASSURANCE/QUALITY CONTROL FOR ANALYSIS OF GENERAL INORGANIC PARAMETERS

The main objective of the rigorous QA/QC requirements incorporated in the EPA Protocols is to assure the production of data of known quality which will meet the client's needs whether it be to support a remedial decision or an enforcement action. The high level of documentation required in conjunction with the built-in QA/QC serves to provide the laboratory or the regulatory agency with legally defensible data. The QA/QC aspects contained within the EPA Methodologies for general inorganic parameters may be classified under the heading method performance QA/QC for the analysis of general inorganic parameters.

Method Performance QA/QC for Analysis of General Inorganic Parameters

- Method Blanks - The analysis of method blanks provides the laboratory and the regulatory agencies a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. The method blank is often used to zero the instrument in spectrophotometric analysis. At least one method blank, consisting of deionized water, must be processed through the entire analytical procedure for every batch of ten samples. Documentation of the results of these method blanks must be included with analytical results in the bound

laboratory notebook. The data must also be entered into the data base of the laboratory information management system.

- Duplicates - Every batch of ten samples for each general inorganic parameter must include one sample analyzed in duplicate. If the results of the two samples do not fall within the range of ±10% for agreement, the batch of samples should be considered suspect and if an exact problem can not be determined, the entire batch of samples must be reanalyzed.

- Spikes - Every batch of ten samples for each general inorganic parameter must include one sample that is spiked with a known concentration of the analyte that is being tested. If the results of the spikes do not fall into a percent recovery range of 85 to 110%, the samples from that batch must either be flagged for matrix effects, or the entire batch of samples must be reanalyzed. The sample batches must be divided into groups of a similar matrix for the spike recovery data to be applicable to the entire batch.

- Initial calibration - The objective in establishing compliance requirements for satisfactory calibration is to insure that the analyst and equipment is capable of producing acceptable quantitative data. A minimum of three standards must be used to determine a calibration curve for these wet and general chemistry parameters. The response of these standards must be documented before any sample batches are processed. Calibration curves must be included in the laboratory book documentation for analysis.

- Continuing calibration check - The calibration must be verified during the analysis of several batches of samples for a given parameter after every 10 samples.

- End standard check - Each sample analysis must end with an end standard check to verify that the validity of the analytical results has carried through to the end of the procedure. Documentation of this end standard must be included in the laboratory notebook account of the sample analysis.

QUALITY ASSURANCE/QUALITY CONTROL FOR ANALYSIS OF SEMI-VOLATILE ORGANICS

The level of QA/QC required by the EPA 600 Series Methodologies, the SW-846 Methodologies, and the EPA CLP Program are very stringent. These requirements assure that the data produced are of a known quality and can be related to Data Quality Objectives. Data Quality Objectives are qualitative and quantitative statements developed by data users to define the quality of the data needed for a particular data collection activity, to support specific remedial decisions or regulatory actions. The QA/QC aspects for organic analyses may be divided into two categories: (1) instrument or system performance QA/QC and (2) method performance or sample QA/QC.

Instrument Performance QA/QC for Analysis of Semi-Volatile Organic Parameters

- Prior to analysis of any blanks or samples by GC/MS, and simultaneous with or prior to analysis of standards, the ion criteria for decafluorotriphenyl- phosphine (DFTPP) must be met as defined in EPA Method 625 or SW-846 Method 8250. The DFTPP tune must be successfully demonstrated every 12 hours of analysis time.

- Three-point initial calibrations (at a minimum, preferably five-point calibrations, which are required for EPA Contract Laboratory Work) at designated concentrations are required prior to any analysis of blanks or sample to define the dynamic range of every GC/MS system. Certain compounds have been selected as System Performance Check Compounds (SPCC) in the EPA Methodologies and must have a minimum average response factor (RF) of 0.05. Other compounds have been designated as Calibration Check Compounds (CCC) by the EPA Methodologies and must have a percent relative standard deviation (% RSD) of less than 30% to ensure the validity of the initial calibration. Only when these SPCC and CCC requirements are met is the instrument calibration considered valid. Refer to the procedure for extraction and analysis by GC/MS of base/neutral and acid extractables in the General Engineering Laboratories Procedures Manual for Organic Chemistry for the compounds required for checking the performance and calibration.

- After 12 hours of analysis, a continuing calibration (at a designated concentration according to the EPA Method 625 or SW-846 Method 8250) must be performed following a successful DFTPP tune. Again, the minimum RF allowed is 0.05 for SPCCs. For CCCs, the RF must not deviate more than 25% from the RF of the initial calibration.

Sample QA/QC for Analysis of Semi-Volatile Organic Parameters

- Method blanks must be analyzed for every 20 samples or in smaller batches of samples, where applicable. A batch of samples can be defined here as a group of samples from a given site over a given time period), whichever is less, for each matrix in a batch, and for each level of concentration. Common phthalate esters must not appear in the blank at greater than five times the required detection limit. All other semi-volatile target compounds must not be present in the blank at greater than the required detection limit.

- Surrogate spike compounds are added to each sample prior to sample preparation at designated concentrations to determine if there is a problem caused by sample preparation, analysis, or matrix. In some cases, especially in the EPA Contract Laboratory Program requirements, required recoveries are defined in the contract. In these cases, if surrogate recoveries do not meet contractully required recovery windows, samples must be re-extracted and re-analyzed.

- Matrix spikes are added to a given sample in duplicate for each matrix and concentration level in a batch of samples. Matrix spike recoveries provide some information on the suitability of the method for the sample matrix but should not be used to determine data usability for other than the given sample unless other information is take into account. Matrix spike recovery information would ideally provide method precision information (its primary objective historically). However, the inability to exercise precision in sampling typical hazardous site samples (largely non-homogenous and difficult to homogenize) has made it impractical to define recovery windows that are required minimums. However, there are recommended performance based QC limits for both relative percent difference (RPD) and percent recoveries of the matrix spike compounds in the EPA Methodologies. If a large deviation from these guidelines occurs, perhaps the method is unsuitable for the given matrix or there is a laboratory bias that must be monitored and addressed if chronic.

The use of rigid analytical protocols, intense QA/QC and data reporting uniformity has successfully shown that these rigid EPA Methodologies and QC programs can and do produce high quality data that can be interpreted and translated into information for use in remedial actions and/or in enforcement of the "liability" provisions of CERCLA when "responsible parties" are identified.

QUALITY ASSURANCE/QUALITY CONTROL FOR ANALYSIS OF VOLATILE ORGANICS

The level of QA/QC required by the EPA 600 Series Methodologies, the SW-846 Methodologies, and the EPA Contract Laboratory Program are very stringent, but they assure that the data produced are of a known quality and can be related to Data Quality Objectives. Data Quality Objectives are qualitative and quantitative statements developed by data users to define the quality of the data needed for a particular data collection activity or to support specific remedial decisions or regulatory actions. The QA/QC aspects for volatile organic analyses may be divided into two categories: (1) instrument or system performance QA/QC and (2) method performance or sample QA/QC.

Instrument Performance QA/QC for Analysis of Volatile Organic Parameters

- Prior to analysis of any standards, blanks, or samples by GC/MS, the ion criteria for 4-bromofluorobenzene (BFB), established in the EPA Method 624 or the SW-846 Method 8240, must be met. The BFB tune must be successfully demonstrated every 12 hours of analysis time.

- Three-point initial calibrations (at a minimum, preferably five-point calibrations, which are required by EPA Contract Laboratory Program) at designated concentrations are required prior to any analysis of blanks or samples to define the dynamic range of every GC/MS system. Certain compounds have been selected as System Performance Check Compounds (SPCC) in the EPA Methodologies and must have a minimum average response factor (RF) of 0.3 (except for that of bromoform which must be 0.25). Other compounds have been designated as Calibration Check Compounds (CCC) by the EPA Methodologies and must have a percent relative standard deviation (% RSD) of less than 30% to ensure the validity of the initial calibration. Only when these SPCC and CCC requirements are met is the instrument calibration considered valid. Refer to the procedure for analysis of volatile organics by GC/MS in the Organic Procedures Manual for General Engineering Laboratories for the compounds that are specified as check compounds for performance and calibration.

- After 12 hours of analysis, a continuing calibration (at a designated concentration according to the EPA Method 624 or SW-846 Method 8240) must be performed following a successful BFB tune. Again, the minimum RF allowed is 0.3 (0.25 for bromoform) for SPCCs. For CCCs, the RF must not deviate more than 25% from the RF of the initial calibration.

- Three-point initial calibrations at designated concentrations are required prior to any analysis of blanks, spikes, or samples when analyzing volatile organics by GC/PID/Hall as described in EPA Methods 601/602. Periodic verification of this calibration must be documented. This should generally be checked and documented every 20 samples.

Sample QA/QC for Analysis of Volatile Organic Parameters

- Method blanks must be analyzed for every 20 samples or in every batch of samples. A batch of samples can be defined here as a group of samples from a given site over a given time period, whichever is less, for each matrix in a batch, and for each level of concentration. Volatile organic target compounds must not be present in the blank at greater than the required detection limit.

- Surrogate spike compounds are added to each sample prior to sample preparation at designated concentrations to determine if there is a problem caused by sample preparation, analysis, or matrix. In some cases, especially in the EPA Contract Laboratory Program requirements, minimum recoveries are defined in the contract. In these cases, if surrogate recoveries do not meet contractully required recovery windows, samples must be re-analyzed.

- Matrix spikes are added to a given sample in duplicate for each matrix and concentration level in a batch of samples. Matrix spike recoveries provide some information on the suitability of the method for the sample matrix but should not be used to determine data usability for other than the given sample unless other information is take into account. Matrix spike recovery information would ideally provide method precision information (its primary objective historically). However, the inability to exercise precision in sampling typical hazardous site samples (largely non-homogenous and difficult to homogenize) has made it impractical to define required recovery windows. However, there are recommended performance based QC limits for both relative percent difference (RPD) and percent recoveries of the matrix spike compounds. If a large deviation from these guidelines occurs, perhaps the method is unsuitable for the given matrix or there is a laboratory bias that must be monitored and addressed if chronic.

- Analysis of a deionized water spike must be analyzed in every batch of ten samples when analyzing sample for volatile organics by GC/PID/Hall as described by EPA Method2 601/602.

The use of rigid analytical protocols, intense QA/QC and data reporting uniformity has successfully shown that these rigid programs can and do produce high quality data that can be interpreted and translated into information for use in remedial actions and/or in enforcement of the "liability" provisions of CERCLA when "responsible parties" are identified.

QUALITY ASSURANCE/QUALITY CONTROL FOR ANALYSIS OF PESTICIDES

The implementation of a rigid QA/QC requirement is an important part of any method. The QA/QC requirements of EPA protocols are necessary to assure that the data collected are of known quality, to allow interlaboratory comparison of data to facilitate including all data in a national data base of environmental contamination, and to ensure that all data collected are legally defensible. The QA/QC program of the EPA protocol has two aspects: (1) requirements for instrument performance and (2) requirements for method performance.

Instrument Performance QA/QC for Analysis of Pesticides

- Prior to analysis of any standards, blanks, or samples, the laboratory must run a three-point initial calibration of all single component analytes and a single point determination fo all multiple component analytes. For each single component analyte, the mean deviation for the three absolute retention times must be less than 0.3 percent of the average of the retention times and the relative standard deviation of the response factors must be less than or equal to 20% of the average of the three determinations.

- All multicomponent analytes must be run periodically during a run sequence to confirm that the initial calibration of those compounds is still valid. Toxaphene, chlordane, and Aroclors 1016, 1242, 1248, 1254, 1260, and 1262, must be run every week. Aroclors 1221 and 1232 must be run monthly. If the combined response factor of any of the multicomponent analytes is more than 20 percent different from the initial calibration value, the GC system is considered out of control and appropriate corrective action must be taken. After the corrections are made, the initial calibration must then be rerun and all samples with detectable levels of PCB's run since the last acceptable set of standards must be rerun.

- Analyte retention time, calibration, resolution, and stability must be monitored every 12 hours by injection of the six compounds listed in Table 2 below.

Table 2 - Performance Evaluation Mixture for Pesticides

Pesticide	Concentration
Beta-BHC	2.5 ng/mL
Gamma-BHC	2.5 ng/mL
Aldrin	25 ng/mL
p,p'-DDT	500 ng/mL
Endrin	50 ng/mL
Dibutylchlorendate	250 ng/mL

For each analyte, the retention time relative to the internal standard dibutylchlorendate must be within 1.5 percent of the average of the calibration standard and the response factor must be within 20 percent of the calibration average. Endrin and DDT are included as analytes that have the potential to degrade on the GC column. Their combined breakdown must be less than 20 percent. Beta-BHC and gamma-BHC are included as resolution checks. The

height of the valley between the two BHC peaks must be no more than 10 percent on either capillary or packed columns. At least one acceptable evaluation standard must be run every 12 hours. If an acceptable evaluation standard cannot be run, the chromatographic system is considered out of control and appropriate correction must be made. After the corrections are made, the initial calibration must be rerun before any sample data are collected. Any data that are not bracketed with acceptable evaluation runs are not acceptable. Those samples must be reinjected.

Sample QA/QC for Analysis of Pesticides

- Blanks - A method blank must be analyzed for every 20 samples extracted by each of the extraction procedures. An acceptable method blank has no analyte present at a level greater than the required detection limit. If an unacceptable method blank is run, all samples analyzed since the last acceptable method blank must be reextracted and reanalyzed.

- Instrument blanks - Instrument blanks are required for the GC. An instrument blank is run by analyzing an injection of solvent. An acceptable instrument blank must have no analyte present at greater than one-half the required detection limit. If an unacceptable instrument blank is run, corrective action must be taken and all samples run since the last acceptable instrument blank must be reanalyzed.

- Matrix Spike/Matrix Spike Duplicate Analysis - A matrix spike/matrix spike duplicate (MS/MSD) analysis must be performed for every 20 samples prepared by each extraction procedure. The MS/MSD pair is a replicated sample prepared in the laboratory by adding matrix spike solution as specified in the EPA Method 608 or the SW-846 Method 8080, to duplicate aliquots of a sample prior to extraction. The MS/MSD pair are extracted, cleaned up, and analyzed according to the EPA Methodologies specified above. The recoveries of the individual compounds that were spiked in the samples are reported in the laboratory notebook documentation for the batch of samples of interest.

- Surrogate Analysis - The surrogate hexabromobenzene (HHB) must be added to all samples and method blanks prior to extraction in order to monitor method performance and sample chromatography. HHB was chosen because it chromatgraphs near the middle of the analytical run, it has a different retention time from commonly observed matrix interferences and hazardous substance list pesticides, and it does not undergo ester hydrolysis unlike the present surrogate, dibutylchlorendate.

- Blank spikes - Ten percent of all sample batches must be a blank spike. The blank spike consists of either laboratory pure water for a batch of water samples or sand for a batch of solid samples. The blank is spiked with a laboratory defined spiking solution. The percent recoveries for these compounds in the spiking solution must be reported in the laboratory notebook documentation for the batch of samples of interest.

MISCELLANEOUS QUALITY ASSURANCE/QUALITY CONTROL CHECKS

The following Quality Control checks may be applicable to many analyses; some of

these consideration may be method-specific.

- Interlaboratory Split Samples - It is a very common practice for the regulatory agencies, such as the Environmental Protection Agency or the South Carolina Department of Health and Environmental Control, to require that samples be split in the field during the collection process, to be analyzed by both the independent commercial laboratory and the laboratory assocatied with the particular regulatory agency. Carefuly comparison studies are made on these split sample results by both the independent laboratory and the regulatory agency. The only true comparison of the data generated from a split sample analysis are when the two laboratories use the same instrumentation, the same methodologies for preparation and analysis, and follow the same Quality Control regimen.

- Intralaboratory Split Samples - On occasion, it is necessary to split a single sample and identify this sample with two different identifications and submit the two samples to the laboratory as individual samples. This "blind" method of submitting the samples to the laboratory eliminates the bias that might occur if the laboratory realized that these two samples were the same. This type of sampling is used as a Quality Control Check for the sample preparation, the instrumental analysis, and the Quality Control procedures.

- Control Charts.- Standard Shewhart Control Charts are plotted for precision and accuracy for all metals, general inorganic parameters, and all organics for:

  + Matrix Spikes
  + Blank Spikes
  + Duplicates

  These Control Charts are set up with both Warning Limits and Control (Action) Limits. The Warning Limits are determined at two times the standard deviation of the recovery (accuracy) or true value (precision) for a minimum of 20 points. The Control Limits are determined at three time the standard deviation of the recovery (accuracy) or true value (precision) for a minimum of 20 points. Both limits should be updated when either 20 more points are accumulated or at the beginning of each quarter, whichever happens first.
  The details of the Control Charts generated by General Engineering Laboratories is discussed in detail in Section XIII of this Quality Control Manual.

- Field Blanks - Field blanks are prepared in the glassware preparation area of the laboratory. Field blanks are prepared for each class of compounds that are to be collected within a batch of samples. For every set of 10 or less volatile organic samples that are collected from a single site, a field blank must be carried to the point of collection. These field blanks are logged in the laboratory with a sample identification number and processed as a regular sample. The data is used to determine possible sources or contamination from the collection process or to confirm that the collection of the samples transpired with no unusual events.

- Quality Control Samples - Quality Control Samples are also called EPA Refernece Samples. The QC Samples are obtained from the United States Environmental Protection Agency, Quality Assurance Branch, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, 26 West St. Clair Street, Cincinnati, Ohio 45268. The telephone number for this laboratory is (513) 569-7325. This laboratory will accept phone orders for QC Samples that need to be ordered. The order will be processed and delivered to our laboratory

within four to six weeks. A record of QC Samples ordered and received must be maintained in the Standard Log Book for both inorganics and organics. A limit of six samples for each parameter are allotted to our laboratory on an annual basis. The most recent QC Samples that are available through this office and the order form for these samples is added to this Quality Control Manual as Appendix 9. These EPA QC Samples are analyzed on a quarterly basis for all parameters for which they are available. The QC Samples are also analyzed with every batch of EPA and other certification audit samples.

- Internal Standards - Internal standards are used in specific analyses according to the requirements of the EPA 600 Series Methodologies and the Procedures outlined in the EPA SW-846 Methodologies. Methods where the use of internal standards for quantitation is mandatory include the analysis of base/neutral and acid extractables by GC/MS, the analysis of volatile organics by GC/MS, the analysis of some compounds by GC, and occasionally in the analysis of metals by ICP or AA or Furnace Techniques. Refer to the General Engineering Laboratories Procedures Manuals for both Inorganics and Organics for the details on the use of internal standards in these methodologies.

- Calibration Standards - Calibration Standards are obtained either from the EPA Repository of Pesticides and Industrial Chemicals (MD-8), Research Triangle Park, North Carolina 27709 USA. The standards obtained from this facility are in the neat form. The phone number for this facility is (919) 541-3951. The emergency number is (702) 798-2690. This facility will not accept phone orders except in the event of an emergency. They will offer technical assistance for standards and methodologies over the phone. The standards to be ordered are selected for the EPA Document: Analytical Reference Standards and Supplemental Data: The Pesticides and Industrial Chemicals Repository/ EPA-600/4-84-082. A copy of the order blank is added to this QC Manual as Appendix 8.
  Pre-wieghed Standards at ppm concentrations are also available to our laboratory from the United States Environmental Protection Agency, Quality Assurance Branch, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, 26 West St. Clair Street, Cincinnati, Ohio 45268. The telephone number for this laboratory is (513) 569-7325. This laboratory will accept phone orders for QC Samples that need to be ordered. The order will be processed and delivered to our laboratory with four to six weeks. A record of standards ordered and received must be maintained in the Standard Log Book for both inorganics and organics. A limit of six standards for each parameters are allotted to our laboratory on an annual basis. The most recent standards that are available through this office and the order form for these standards is added to this Quality Control Manual as Appendix 9.
  Certified standards are also used by our laboratory from the following sources:

  Chem Service Inc.
  PO Box 3108
  West Chester, Pennsylvania 19381

  Supelco Inc.
  Supelco Park
  Bellefonte, Pennsylvania 16823-0048

Aldrich Chemical Company
940 West Saint Paul Avenue
Department 700
Milwaukee, Wisconsin 53233 USA

Sigma Chemical Company
PO Box 14508
St. Louis, Missouri 63172 USA

American Scientific Products
8350 Arrowbridge Blvd.
Charlotte, North Carolina 28210

Fisher Scientific Company
Department 301276-01
PO Box 405
Pittsburgh, Pennsylvania 15230

Only the highest grade of standards available are purchased for use in our laboratory. The standards, when purchased in the neat form, are certified to have a known purity. If purchased in a pre-weighed or pre-diluted form, the solutions are certified to be of a given concentration. When either neat standards or certified solutions are received in the central supply area, the container is marked as received and dated with the date of that day.
When standards are prepared in stock, intermediate, and working concentrations, either from the neat form or from a certified solution, the preparation of that standard is documented in a bound Standard Log Book. There are separate volumes of the Standard Log Book for:

- Metals
- General Inorganics
- Volatile Organics
- Pesticides/Herbicides/PCBs/Phthalate Esters
- Base/Neutral and Acid Extractables
- General Organics, including Hazardous Waste Characterization Standards for Organic Analyses

The forms used to document the preparation of stock, intermediate, and working standards are added to this QC Manual as Appendix 9.
Holding times for standards vary. Expiration dates are given on both the EPA standards and the standards that are purchased from the vendors. No standard is ever used after the expiration date. Standards that are in solution are meniscus-marked and periodically checked to insure that no concentration of the solvent has occured. If there is evident concentration of the solvent medium, the standard solution is discarded and a replaced with a freshly prepared standard solution.

SECTION XII

ROUTINE PROCEDURES USED TO ASSESS PRECISION AND ACCURACY

XII. ROUTINE PROCEDURES TO ASSESS DATA PRECISION AND ACCURACY

The routine procedures used to assess data for precision, accuracy, and completeness involve the generation and monitoring of QC Control Charts for accuracy, precision, and completeness. Thorough data review by the Quality Control Officer is necessary for these aspects of QC to be properly monitored.

Precision refers to the agreement or reproducibility of a set of replicate results among themselves without assumption of any prior information as to the true result. Precision is usually expressed in terms of the deviation, variance, or range. Accuracy is the nearness of a result or the mean of a set of results to the true value. Accuracy is usually expressed in terms of error, bias, or percentage recovery.

Good precision often is an indication of good accuracy, but one can obtain good precision with poor accuracy if a systematic (determinate) error is present in the method used. Systematic errors are either positive or negative in sign. The other general classification of errors in analysis is indeterminate (random) errors. These are errors inherent in the analytical methods because of uncertainties in measurements. An example is the measurement of the height of a gas chromatographic peak with a ruler, which even the most careful analyst can measure only to the nearest one mm. Indeterminate errors are random, that is, they are just as likely to be positive as negative. For this reason, the average of several replicate measurements is always more reliable than any of the individual measurements. Although random errors are unavoidable, determinate errors can be corrected once their cause is located.

Absolute error is the difference between the experimental result and the true value. Relative error is absolute error divided by the true value and multiplied by 100 to yield percent relative error or by 1000 to yield parts per thousand relative error. Bias is defined as the mean of the differences (having regard for signs) of the results from the true value.

In addition to recording numerical results of each analysis of an internal QC sample, it is necessary to construct a Quality Control Chart. A separate chart must be generated for both Accuracy Data as well as Precision Data. The purpose of this chart is to provide graphic assessment of accuracy and precision for the analysis of each substrate and instant detection of erroneous data. The charts allow quick and easy observation of recovery trends for a particular analysis and have long term value for the self evaluation of analytical output by staff personnel. Another significant value of the charts is that of providing a laboratory administrator with a rapid assessment of the continuing analytical capability of the staff chemists as related to the output of valid analytical data.

The applicability of control chart technics is based on the assumption that laboratory data approximate a normal distribution. The data from such a system, however, can be presented in a different graphic way by plotting on the vertical scale the units of the test results and on the horizontal scale the order or sequence in which the results were obtained. The mean and limits of dispersion in terms of the standard deviation are calculated and plotted as in the control chart in Figure 1.

For the best results make at least 20 determinations on a sample before calculating the standard deviation or plotting the control chart. All results need not be obtained on the same day; optimally accumulate them as part of a day-to-day operation. A result may be obtained on a control sample each time an unknown or group of unknowns is analyzed.

For example, consider a collection of results, in milligrams per liter, obtained by analysis of a water sample for copper, as follows:

The mean of this series is 0.256 mg/L, the standard deviation is 0.020 mg/L, and the resulting control chart is that of Figure 2.

The upper and lower control limits (UCL and LCL) are at +3 and -3 standard deviations from the mean, respectively, and the upper and lower warning limits (UWL and LWL) at +2 and -2 standard deviations.

This is a control chart for individuals, and is used most frequently in chemical analysis. Laboratory control requires adding known or standard samples to the unknown samples, thus increasing both the cost and time or analysis.

If a result falls outside the control limits on an individual control chart, the analysis is said to be "out of control"; take immediate action to determine the cause of the outlying result. Consider unreliable any analytical results obtained for samples on the same day as the erroneous result occurred on the known sample and repeat the analyses after corrective action has been taken and the procedure is back in control. It sometimes is desirable to take milder action when the results exceed the warning limits, a because an unduly high percentage of results exceeding these limits is a warning that laboratory precision may not be as good as expected or that distribution of results is not normal.

Accuracy in analytical chemistry is a measure of the difference between the mean of the results and the true value. Most frequently, accuracy is expressed in terms of the mean error:

$$\text{mean error} = \text{mean} - \text{true value.}$$

Accuracy can be determined only if the control sample is prepared in the laboratory from known amounts of pure regents. The accuracy of the determination is the difference between the mean of the 20 determinations(collected for the construction of the initial control chart) and the true value or known amount chemical added.

The true value (T.V.) also can be plotted on the control chart. Usually it falls only slightly above or below the mean. If the true value is more than half way between the mean and the upper or lower warning limit (i.e., the mean error is greater than one standard deviation), check the method, reagent, glassware, technic, or instruments for bias and take corrective action.

Completeness is a measure of the amount of valid data obtained compared to the amount that was expected from a measurement system under normal conditions. For all practical purposes, this measurement is the percentage of analytical results that fall within the upper and lower warning limits of the accuracy control chart.

The formula for % Completeness is:

$$\%\text{Completeness} = (P_{2S}/P_T) \times 100$$

Where:

P_{2S} = Number of observed values for percent recovery outside the 2 sigma range

P_T = Number of total observations for percent recovery

Examples of Quality Control Charts for accuracy and precision from the laboratory records of General Engineering Laboratories are shown in Appendix 10.

SECTION XIII

CORRECTIVE ACTION

XIII. CORRECTIVE ACTION

Limits of Data Quality Acceptance

For routine analytical work and special projects to run smoothly, it is necessary for guidelines to be established for acceptability of data and performance for a plan of corrective action to be implemented.

Corrective action is implemented when one of the following situations occurs:

- Quality Control spike data is out of the established limits of acceptance
- Quality Control duplicate data is out of the established limits of acceptance
- Background or blank data is unusually high or masks area for parameter measurement
- An instrument is not functioning properly
- An analytical procedure is not producing the data that is needed to provide necessary information and the procedure appears to be out of control
- Performance Audit Data evaluation as suspect or not acceptable data
- Quality Assurance and Quality Control problems detected from system audits
- Poor reproducibility of data detected in laboratory or interfield comparison studies
- Problems detected in Quality Assurance audits conducted by the various certification offices

The guidelines for acceptable data are outlined for analysis of metals, general inorganic parameters, semi-volatile extractable organics, volatile organics, pesticides, and polychlorinated biphenyls (PCBs) in Section X of the QC Manual under the heading of Laboratory Quality Control Checks. These quality control acceptance limits are defined in this section for:

- Quality Control spikes
- Quality Control duplicates
- Background blank data
- Instrument calibration data
- Continuing instrument calibration data
- Surrogate spike data
- Internal standard data and response factors

As a general rule, corrective action for Quality Control data is implemented when the following limits are not achieved:

Metals

- Spikes must fall in the 90 to 110% recovery range.
- Duplicates (digested) must fall in the ±10% range.
- Replicate analyses and check standards must fall in the ±4% range.

General Inorganics

- Spikes must fall in the 85 to 115% recovery range.
- Duplicates (digested) must fall in the ±10% range.
- Replicate analyses and check standards must fall in the ±10% range.

Organics

- Spikes must fall in the 75 to 125% recovery range.
- Duplicates (digested) must fall in the ±20% range.
- Replicate analyses and check standards must fall in the ±10% range.

Acceptance limits for data quality are also outlined in Section IV of this Quality Control Manual under the heading of QA/QC Objectives.

Procedures for Corrective Action

When it is established that a plan for corrective action must be implemented, necessary step must be taken to rectify the problem situation. There are various procedures for corrective action plans, depending on the problem that has occurred.

Typical Corrective Action plans include:

- Recalibration of the measurement instrument if tuning, calibration, or continuing calibration criteria are not achieved.
- Re-extraction and re-analysis of sample batches where the Quality Control spikes, duplicates, surrogates, or internal standards were not within acceptable limits of quality.
- Work order called in to the instrument vendor if the instrument has failed or is not working properly to produce data of acceptable quality.
- Review of the analysts analytical techniques and philosophies if a problem is detected that seems to be unrelated to instrument or methodology trouble.
- The analysis of an EPA-certified reference standard if analyte recovery problems cannot be easily defined.

A plan of Corrective Action may be decided upon at the instrument operator's level. This plan may also be implemented at this level. The plan should, however, be presented to the group leader of the section and to the laboratory manager. The laboratory manager must notify the project manager when problems have arisen and corrective action is in order. All corrective action plans should be submitted to the Quality Control Officer for documentation in the Quarterly Quality Control Report. The Quarterly Quality Control Report will insure that the owners and the full management staff of General Engineering Laboratories are aware of the kinds of problems that resulted in corrective action that occurred during the quarter.

Corrective Action Resulting from On-Site Audits

The nature of the on-site audit for the certification programs is to detect any deficiencies in the overall operation of the laboratory, including methodologies, instrumentation, personnel, and data handling/storage. If corrective action is deemed necessary by any of the certification officers, it is the policy of General Engineering Laboratories to meet the full requirements of the corrective action in the specified time frame of the certification agency.