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QUALITY ASSURANCE PROJECT PLAN FOR BEULAH LANDFILL SITE NAS PENSACOLA  
FL  
2/1/1992  
ENGINEERING-SCIENCE

**QUALITY ASSURANCE  
PROJECT PLAN**

**BEULAH LANDFILL SITE**

PREPARED FOR

**BOARD OF COUNTY COMMISSIONERS  
ESCAMBIA COUNTY  
SOLID WASTE MANAGEMENT  
CANTONMENT, FLORIDA**

PREPARED BY

**ENGINEERING-SCIENCE**

Atlanta, Georgia

FEBRUARY 1992  
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ENGINEERING-SCIENCE

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**PREPARED BY**

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**MARCH 1992**

**Reviewed and approved by:**



**Project Manager**



**Quality Assurance Manager**

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## SECTION 1 PROJECT DESCRIPTION

### 1.1 PURPOSE

This Quality Assurance Project Plan (QAPP) has been developed for use during sampling and analytical activities at the Beulah Landfill site to ensure appropriate sample analysis. The sampling program for soils, groundwater, surface water, and sediments is designed to confirm the magnitude and extent of contamination at the site and to characterize the nature of any contaminants detected. The QAPP serves as a controlling mechanism during the investigation to ensure that all data collected are valid, reliable, and defensible in a court of law. This document outlines the organization, objectives, and quality assurance/quality control (QA/QC) activities necessary to achieve the desired data quality goals.

### 1.2 SITE BACKGROUND

The Beulah Sanitary Landfill construction and operation began in 1966 over a 101.9-acre site. Approximately 90 acres of the site was utilized for landfilling with the remaining 11 acres consisting of Coffee Creek and perimeter ditches. The site is located at the north end of Jamesville Road in Section 15, Township 1S, Range 31W at latitude 30 30' 8"N and longitude 87 20' 42"W (Figure 1.1).

Solid waste was first deposited in the southwest section at an average depth of 15 to 20 feet. As the disposal cells moved to the east, the depths increased to 30 to 35 feet. In 1968, the first domestic septage and wastewater treatment sludges were deposited in an excavated and bermed area (approximately 10 acres) at the southwest corner. In 1976, the sludge holding pond was filled with construction demolition debris and solid waste and covered with 12 inches or more of on-site material. In November 1977, an area of approximately 20 acres was constructed and diked to receive liquid sludges in the eastern portion of the property. This area was previously filled with solid waste that absorbed the liquid sludge to create a solid/spongy mixture. All sludge dumping was ceased in June 1984.

Records of the waste stream entering the Beulah Landfill facility were destroyed by the Superior Court of Escambia County and no composition studies have been done to date. Based on operator memory, the waste stream consisted of a combination of solid and liquid wastes from residential, commercial, construction,

and demolition as well as industrial sources including wastes from local military bases. The major waste depositors included the franchise collectors and haulers, private haulers, and the general public. Liquid waste haulers deposited septic wastes, wastewater treatment plant sludges, commercial and industrial sludges, and liquid waste in ponds onsite. All disposal activities were concluded in 1984.

A more detailed discussion of the site can be found in Section 2 of the Work Plan.

### **1.3 SAMPLING OBJECTIVES**

The objective of the field sampling program at Beulah Landfill is to acquire additional data needed to confirm the presence or absence of contamination at the site, characterize the principal pathways to potential receptors, and identify sources. Sampling efforts will include groundwater, surface water, soil and air sampling. Soil sampling will be done to characterize the amounts and types of contaminants in the old sludge ponds. This information will be necessary to evaluate potential risks to human and environmental receptors. Groundwater and surface water sampling will be conducted to characterize water quality. The chemical analysis results from these samples will be used to confirm presence or absence of contaminants to determine the horizontal and vertical extent of contamination, and to determine the risks posed by contaminants that may occur in these waters. Air sampling will be performed to characterize the air pathways for potential risks to on-site as well as off-site receptors. Table 1.1 provides a summary of field activities to be completed at the Beulah Landfill site.

### **1.4 SAMPLE LOCATION, FREQUENCY, AND RATIONALE**

The sample location, frequency, and rationale are described in detail in Section 4 of the Sampling and Analysis Plan.

### **1.5 DATA QUALITY OBJECTIVES**

Data Quality Objectives (DQOs) are qualitative and quantitative statements that specify the quality of the data required to support decisions made during RI/FS activities and are based on the end uses of the data to be collected. As such, different data uses may require different levels of data quality. There are five analytical levels that address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. The levels are as discussed below.

#### **1.5.1 Screening (DQO Level 1)**

This provides the lowest data quality, but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to ARARs, initial site characterization to locate areas for subsequent and more accurate

analyses, and for engineering screening of alternatives (bench-scale tests). These types of data include those generated onsite through the use of photoionization detectors, pH, conductivity, temperature, and other real-time monitoring equipment at the site.

#### **1.5.2 Field Analyses (DQO Level 2)**

This provides rapid results and better quality than in Level 1. This level may include mobile lab generated data depending on the level of quality control exercises. No analyses will be performed at Level 2.

#### **1.5.3 Engineering (DQO Level 3)**

This provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab generated data and some analytical lab methods (e.g., laboratory data with quick turnaround used for screening, but without full quality control documentation). The parameters that require Level 3 analysis include:

- Dissolved Oxygen (EPA 360.1)
- Dissolved Residue (EPA 160.1)
- Turbidity (EPA 180.1)
- Chloride (EPA 9250)
- Fluoride (EPA 340.2)
- Sulfate (EPA 9038)
- Nitrate (EPA 352.1)
- Nitrite (EPA 354.1)

#### **1.5.4 Confirmational (DQO Level 4)**

This provides the highest level of data quality and is used for purposes of risk assessment, evaluation of remedial alternatives, and PRP determination. These analyses require full CLP analytical and data validation procedures in accordance with EPA recognized protocol. The analyses to be performed at Level 4 include:

- Metals (CLP SOW)
- Volatile Organics (CLP SOW)
- Semivolatile Organics (CLP SOW)
- Pesticides/PCBs (CLP SOW)

### **1.5.5 Non-Standard (DQO Level 5)**

This refers to analyses by non-standard protocols. For example, when exacting detection limits or analysis of an unusual chemical compound is required, this analyses will be utilized. These analyses often require method development or adaptation. The level of quality control is usually similar to DQO Level 4 data. Level 5 analyses will not be used during this investigation.

### **1.6 PROJECT SCHEDULE**

Samples will be collected and analyzed upon approval of the Work, Health and Safety, Sampling and Analysis, and Quality Assurance Plans. The project schedule is presented in Section 8 of the Work Plan.

**TABLE 1.1**  
**SUMMARY OF FIELD ACTIVITIES**

<b>Field Activity</b>	<b>Purpose</b>	<b>Sampling Media</b>	<b>Analyses</b>
Measure surface water flow and sample surface water and sediments at up to eight locations.	Confirm previous results and further characterize surface water quality.	Surface water and sediments	Full TCL/TAL scan with Level IV DQO.
Collect up to 27 surface soil samples.	Characterize potential contamination sources for direct exposure pathway.	Soil	Full TCL/TAL scan with Level IV DQO.
Sample up to 12 monitoring wells.	Confirm previous results and further characterize groundwater quality.	Groundwater	Full TCL/TAL scan with Level IV DQO.
Collect ambient air samples at up to four locations.	Characterize air quality.	Air	Respirable particulates, TCL/TAL analysis of particulates, VOCs by EPA Method TO14-1.
Perform Rapid Bioassessment Protocol II.	Determine biota/habitat of streams.	Bottom sediments/ benthic organisms.	100 organism subsamples with Taxonomic ID to family level.



## **SECTION 2 PROJECT ORGANIZATION AND RESPONSIBILITIES**

### **2.1 PROJECT TEAM**

The Contractor team for this RI/FS project includes:

- PRP Oversight.
- Project Manager.
- Field Operations Manager.
- Subcontractor Services.

Figure 2.1 presents the overall project organization. Subcontractors will be utilized for site preparation, and surveying services. Site sampling work will be performed by the University of West Florida, Wetlands Research Laboratory. Laboratory services will be provided by Kiber Analytical Services, Atlanta, Georgia.

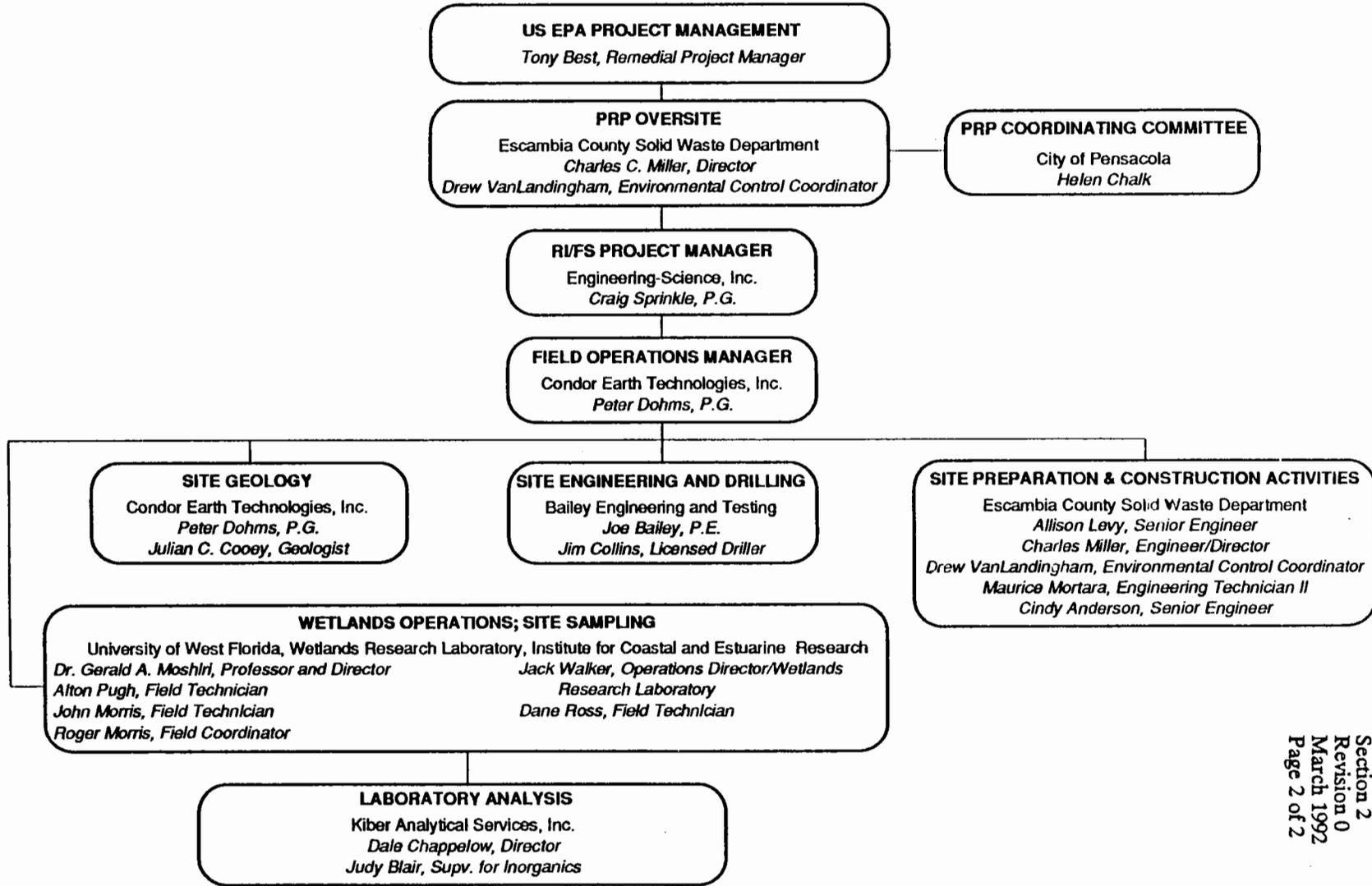
### **2.2 RESPONSIBILITIES OF PROJECT PERSONNEL**

Mr. Craig L. Sprinkle, P.G., will serve as the project manager. He is responsible for directing the progress of the work and for providing technical review for all project deliverables.

QA, including analytical data validation, will be under the direction of the ES QA Officer, Mr. Douglas Chatham.

Mr. Peter Dohms, P.G., will be responsible for supervising all field activities. He is responsible for insuring that all the procedures and field QA requirements described in the project SAP are followed. He will also serve as the site health and safety officer and will insure that the procedures contained in the project H&S plan are followed.

# PROJECT ORGANIZATION REMEDIAL INVESTIGATION AND FEASIBILITY STUDY (RI/FS) BEULAH LANDFILL SITE



Beulah QAPP  
 Section 2  
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 March 1992  
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Figure 2.1

### SECTION 3

## QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA IN TERMS OF PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS AND COMPARABILITY

The quality assurance objectives for all measurement data include considerations for precision, accuracy, completeness, representativeness and comparability. Parts of this section incorporate by reference the EPA CLP protocols found in the 1988 EPA CLP SOW (as revised through 1989). Analyte-specific quality assurance objectives for precision and accuracy are listed in Tables 3.1 and 3.2 with method detection limits.

### 3.1 PRECISION

The objective for precision for the selected chemical analysis laboratory is to equal or exceed the precision demonstrated for the applied analytical methods on similar samples. Precision is evaluated most directly by recording and comparing multiple measurements of the same parameter on the same exact sample under the same conditions. It is expressed in terms of relative percent difference (RPD) calculated by the method provided in Section 12.

Acceptable levels of precision will vary according to the sample matrix, the specific analytical method, and the analytical concentration relative to the method detection limit. For organic parameters, RPD criteria will be evaluated on samples spiked in duplicate with specified compounds. The spiking compounds to be used and the advisory limits for recovery and RPDs for this project are:

Compound	PR Water	RPD Water	PR Soil	RPD Soil
<b>Volatile Organics</b>				
1,1-Dichloroethane	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-142	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

Compound	PR Water	RPD Water	PR Soil	RPD Soil
<b>Semivolatile Organics</b>				
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
n-Nitroso-di-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36
<b>Pesticides/PCBs</b>				
gamma-BHC (lindane)	56-123	15	46-127	50
Heptachlor	40-131	20	35-130	31
Aldrin	40-120	22	34-132	43
Dieldrin	52-126	18	31-134	38
Endrin	56-121	21	42-139	45
4,4' -DDT	38-127	27	23-134	50

For inorganic analytes, the project limit on RPD is  $\pm 20$  percent if both analyses are greater than the CRQL. If either or both the analysis results are below five times the CRQL, then the RPD is not calculated; instead, the results must agree within  $\pm$  CRQL.

### 3.2 ACCURACY

The objective for laboratory analytical accuracy is to equal or exceed the accuracy demonstrated for the applied analytical methods on samples of similar matrix and concentration of contaminants. Accuracy is determined by analyzing a sample and its corresponding matrix spike sample. Accuracy is expressed as percent recovery (PR) calculated by the method in Section 12.

The degree of accuracy and the recovery of analyte to be expected for the analysis of QA samples and spiked samples is dependent upon the matrix, method of analysis, and compound or element being determined.

Percent recovery guidelines established by EPA will be used to evaluate accuracy and the selected spike compound for organic analyses. Exhibit D of the 1988 CLP SOW lists the recovery guidelines. In addition, for each method, EPA has

selected appropriate surrogate compounds. The CLP limits for surrogate recoveries for this project are:

Compound	PR Water	PR Soil
<b>Volatile Organics</b>		
Toluene-d <sub>8</sub>	88-110	84-138
Bromofluorobenzene	86-115	59-113
1,2-Dichloroethane-d <sub>4</sub>	76-114	70-121
<b>Semivolatile Organics</b>		
Nitrobenzene-d <sub>5</sub>	35-114	23-120
2-Fluorobiphenyl	43-116	30-115
Terphenyl-d <sub>14</sub>	33-141	18-137
Phenol-d <sub>5</sub>	10-110	24-113
2-Fluorophenol	21-110	25-121
2,4,6-Tribromophenol	10-123	19-122
2-Chlorophenol-d <sub>4</sub>	33-110	20-130 (advisory)
1,2-Dichlorobenzene-d <sub>4</sub>	16-110	20-130 (advisory)

### 3.3 COMPLETENESS

Completeness is defined as the percentage of measurements or the amount of data required in order to make a decision concerning a site. The completeness goal is the same for all data uses; that is, that all the data necessary for a valid study be collected. At the end of the data validation process, an assessment of the completeness will be made. If data gaps are apparent, an attempt will be made to collect the required data. Goals of 100% completeness for critical samples and 90% for all other samples per medium must be attained.

### 3.4 REPRESENTATIVENESS

Samples must be representative of the population. Where appropriate, the population will be statistically characterized to express the degree to which the data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process, or an environmental condition.

Sample selection and handling procedures will incorporate consideration of obtaining the most representative sample possible. Representativeness of specific samples will be achieved by the following:

- Collect samples from locations fully representing the site conditions.
- Use appropriate sampling procedures and equipment.

- Use appropriate analytical methodologies for the parameters and detection limits required.
- Analyze within the appropriate holding time.

The field program will include collection of QA samples which will be used to evaluate the representativeness of the analytical data. QA samples to be collected are trip blanks, rinseate blanks, field blanks, field duplicates, matrix spikes (MS) and matrix spike duplicates (MSD). QA samples will not be collected for testing of physical parameters.

**Trip blanks** are HPLC-grade water samples which originate in the laboratory, are transported to the field, and are handled along with the samples collected in the field. They are packaged and shipped to the analytical laboratory with shipment of samples that are to be analyzed for volatile organic compounds (VOCs). The purpose of the trip blanks is to detect contamination introduced into the samples during packaging and shipping. One set of trip blanks will be handled and shipped with each package of samples that are to be analyzed for VOCs.

**Rinseate blanks** are water samples which are prepared in the field by rinsing decontaminated sampling equipment with analyte-free water. The rinseate blanks are analyzed for the same substances as are the samples collected with the associated sampling equipment. The purpose of the rinseate blanks is to detect contamination introduced into the samples through improperly decontaminated sampling equipment. Rinseate samples will be collected every day of sample collection, but initially, only those from every other day will be analyzed.

**Field blanks** are samples of the potable rinse water and of the analyte-free water used for equipment decontamination. The samples are collected in the field and analyzed for the same parameters as the environmental samples which are collected with decontaminated equipment. The purpose of the field blanks is to detect contamination which may be introduced into the samples through decontamination rinse waters. One field blank will be collected from each manufacturer's lot of analyte-free water and from the potable rinse water source.

**Field duplicates** are collected at the same location, using the same sampling method, as an environmental sample. The duplicate is collected immediately after the original environmental sample and is given a fictitious identifier. The purpose of the field duplicates is to evaluate reproducibility of sampling and analytical methods. Ideally, the analytical results from the duplicate will be comparable to those from the original sample. One field duplicate will be collected for every ten environmental samples from each medium.

**MS/MSDs** are collected from the same locations as environmental samples and are spiked with known quantities of organic compounds in the analytical laboratory.

The results are used to evaluate interference effects on the analyses caused by the matrix being analyzed.

### 3.5 COMPARABILITY

All data will be calculated and reported in units consistent with other organizations reporting similar data. The results of analyses can be compared with analyses by other laboratories because the objectives of this project for comparability are:

- To demonstrate traceability of standards to known sources;
- To use standard methodology to the greatest practicable extent;
- To report results from similar matrices in consistent units; and
- To apply appropriate levels of quality control within the context of the Laboratory Quality Assurance Program.

By using traceable standards and standard analytical methodology, the field and laboratory analytical results can be compared to other studies performed similarly. The generic QA/QC Program Plan for Kiber Analytical Services, Inc. (September 1990) documents internal performance evaluations used to document performance compared to other laboratories.

The comparability objective will be achieved when at least 95 percent of the data are collected using standard methods, equipment, and procedures as described herein and in the project Work Plan, and Sampling and Analysis Plan.

**TABLE 3.1  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppm)</b>	<b>Method</b>	<b>Reference</b>
Total Organic Carbon	Water	ID	106,4.9	4	9060	1
Conductivity	Water	5,3.6	97,7.0	0.1	120.1	1
pH	Water	1.8,2.5	97,0.9	ID	9040	1
Temperature	Water	0,0	NA	NA	170.1	1
Oxygen,Dissolved	Water	4.8,2.3	102,35	0.1	360.1	1
Residue, Dissolved	Water	1.3,1.0	103,17	1.0	160.1	2
Turbidity	Water	4,1.2	85,8.1	0.1	180.1	2
Chloride (automated)	Water	0.47,0.21	101,3.5	0.5	9250	2
Fluoride	Water	5,3.6	110,3.6	0.01	340.2	2
Sulfate(Turbidimetric)	Water	2.6,2.1	102,5.6	1.0	9038	2
Nitrogen, Nitrate (automated-Cadmium)	Water	6,4.3	97,2.9	0.1	352.1	2
Nitrogen, Nitrite (automated)	Water	10,5.0	105,3.3	0.1	354.1	2
<b>TAL Metals</b>					<b>CLP-SOW</b>	
Aluminum	Water Soil	5.2, 4.7	92, 3.6	0.03 3.0		4
Antimony	Water Soil	12, 8.6	79, 17	0.043 4.3		4

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppm)</b>	<b>Method</b>	<b>Reference</b>
Arsenic	Water	16, 9.3	71, 9.6	0.003		4
	Soil	12,6.9	10.1	0.6		4
Barium	Water	14, 9.9	76, 0.5	0.003		4
	Soil	10,5.0	100,6.7	0.3		4
Beryllium	Water	1.3, 2.0	96, 0.1	0.001		4
	Soil			0.1		
Cadmium	Water	5.5, 3.0	98, 0.2	0.001		4
	Soil	9,2.2	101,8.1	0.7		4
Calcium	Water	11, 1.7	91, 2.6	0.013		4
	Soil			1.4		
Chromium	Water	4.8, 0.1	95, 3.1	0.008		4
	Soil	10,2.0	89, 8.4	0.8		4
Cobalt	Water	4.0, 3.9	92, 4.5	0.006		4
	Soil			0.6		
Copper	Water	4.1, 1.2	92, 0.3	0.009		4
	Soil	10,2.0	101,5.2	0.6		4
Cyanide	Water	6.9, 2.6	96, 2.5	0.005		4
	Soil			0.125		
Iron	Water	6.0, 0.4	88, 7.0	0.004		4
	Soil			0.4		
Lead	Water	20, 15	84, 15	0.001		4
	Soil	9,6.3	110,10.2	0.2		4

**TABLE 3.1--Continued  
QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppm)</b>	<b>Method</b>	<b>Reference</b>
Magnesium	Water	6.0, 2.9	98, 1.1	0.078		4
	Soil			7.8		
Manganese	Water	3.1, 1.4	94, 0.2	0.001		4
	Soil			0.1		
Mercury	Water	10,7.1	102,3.3	0.00001		4
	Soil	20,16.0	89,6.0	0.001		4
Nickel	Water	5.7, 4.0	95,0.2	0.011		4
	Soil			1.1		
Potassium	Water	8.1, 0.9	86, 36	2.157		4
	Soil			216		
Selenium	Water	21, 10	78, 9.0	0.002		4
	Soil	9,3.8	110,8.2	0.4		4
Silver	Water	23, 9.3	44, 5.1	0.001		4
	Soil	20,1.1	127,0.6	1.0		4
Sodium	Water	20, 2.7	95, 40	0.067		4
	Soil			6.7		
Thallium	Water			0.001		
	Soil			0.2		
Vanadium	Water	5.9, 2.1	96, 2.0	0.010		
	Soil			1.0		
Zinc	Water	8.6, 1.2	93, 0.3	0.003		4
	Soil	12,3.0	103,5.8	0.3		4

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
					CLP-SOW	
Acetone	Water			60		
	Sediment	25,10.0	135,9.9	60		3
Benzene	Water	30,17.4	95,2.0	0.6		3
	Sediment	30,17.4	95,2.0	0.6		3
Bromodichloromethane	Water	15,5.9	103,15.8	0.7		3
	Sediment	15,5.9	103,15.8	0.7		3
Bromoform	Water	15,3.4	118,23.5	0.6		3
	Sediment	15,3.4	118,23.5	0.6		3
Bromomethane	Water	27,13.0	100,10.0	1.1		3
	Sediment	27,13.0	100,10.0	1.1		3
2-Butanone(MEK)	Water			90		
	Sediment	30,14.0	131,20.0	90		3
Carbon disulfide	Water			0.7		
	Sediment	27,15.1	119,18.2	0.7		3
Carbon tetrachloride	Water	12,2.5	110,16.8	1		3
	Sediment	12,2.5	110,16.8	1		3
Chloroethane	Water	17,6.1	121,16.9	10		3
	Sediment	17,6.1	121,16.9	10		3
Chlorobenzene	Water	16,9.0	98,22.8	1		3
	Sediment	16,9.0	98,22.8	1		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
Chloroform	Water	16,2.2	93,3.3	1		3
	Sediment	16,2.2	93,3.3	1		3
Chloromethane	Water	27,21.4	113,18.1	10		3
	Sediment	27,21.4	113,18.1	10		3
Chlorodibromoethane	Water	17,1.8	101,3.0	0.4		3
	Sediment	17,1.8	101,3.0	0.4		3
1,1-Dichloroethane	Water	13,5.0	105,3.6	0.7		3
	Sediment	13,5.0	105,3.6	0.7		3
1,2-Dichloroethane	Water	17,3.2	102,4.5	0.5		3
	Sediment	17,3.2	102,4.5	0.5		3
1,2-Dichloroethene (total)	Water			0.7		
	Sediment	25,7.2	130,10.0	0.7		3
1,1-Dichloroethene	Water	17,10.6	112,6.1	0.9		3
	Sediment	17,10.6	112,6.1	0.9		3
1,2-Dichloropropane	Water	25,3.0	100,10.0	0.6		3
	Sediment	25,3.0	100,10.0	0.6		3
cis-1,3-Dichloropropene	Water	23,8.0	100,10.0	1		3
	Sediment	23,8.0	100,10.0	1		3
trans-1,3-Dichloropropene	Water	25,5.0	100,10.0	0.3		3
	Sediment	25,5.0	100,10.0	0.3		3
Ethylbenzene	Water	14,10.0	98,24.8	0.5		3
	Sediment	14,10.0	98,23.8	0.5		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
2-Hexanone	Water			10		
	Sediment	21,8.1	140,8.7	10		3
Methylene chloride	Water	15,10.7	87,18.8	4.5		3
	Sediment	15,10.7	87,18.8	4.5		3
4-Methyl-2-pentanone				10		
	Sediment	20,11.7	90,6.1	10		3
Styrene				0.7		
	Sediment	27,10.8	95,12.2	0.7		3
1,1,2,2-Tetrachloroethane	Water	16,6.9	93,17.6	0.9		3
	Sediment	16,6.9	93,17.6	0.9		3
Tetrachloroethene	Water	13,1.8	106,6.0	0.6		3
	Sediment	13,1.8	106,6.0	0.6		3
Toluene	Water	15,7.1	98,2.3	1		3
	Sediment	15,7.1	98,2.3	1		3
1,1,1-Trichloroethane	Water	12,1.5	106,7.3	3.4		3
	Sediment	12,1.5	106,7.3	3.4		3
1,1,2-Trichloroethane	Water	14,2.0	95,17.1	0.6		3
	Sediment	14,2.0	95,17.1	0.6		3
Trichloroethene	Water	13,3.6	104,22.7	1		3
	Sediment	13,3.6	104,22.7	1		3
Vinyl acetate	Water			0.7		
	Sediment	25,16.2	129,5.0	0.7		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
Vinyl chloride	Water	30,18.0	100,10.0	10		3
	Sediment	30,18.0	100,10.0	10		3
Xylenes	Water	12,9.0	115,11.0	1.1		3
	Sediment	12,9.0	115,11.0	1.1		3
<b>Base, Neutral and Acid Extractables/Semivolatiles</b>					<b>CLP-SOW</b>	
Acenaphthene	Water	12,1.7	110,9.2	2		3
	Sediment	12,1.7	110,9.2	70		3
Acenaphthylene	Water	10,4.1	100,4.0	2		3
	Sediment	10,4.1	100,4.0	74		3
Anthracene	Water	11,2.2	81,5.1	1.3		3
	Sediment	11,2.2	81,5.1	4.5		3
Benzo(a)anthracene	Water	12,2.3	98,3.2	1		3
	Sediment	12,2.3	98,3.2	35		3
Benzo(b)fluoranthene	Water	12,3.0	99,1.0	1.2		3
	Sediment	12,3.0	99,1.0	42		3
Benzo(k)fluoranthene	Water	16,5.4	104,3.4	0.8		3
	Sediment	16,5.4	104,3.4	28		3
Benzoic acid	Water			32		
	Sediment			1,130		
Benzo(ghi)perylene	Water	12,1.1	98,8.3	0.9		3
	Sediment	12,1.1	98,8.3	32		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
Benzo(a)pyrene	Water	15,6.4	98,8.3	1		3
	Sediment	15,6.4	98,8.3	35		3
Benzyl alcohol	Water	12,3.4	100,2.4	1.5		3
	Sediment	12,3.4	100,2.4	52		3
Bis(2-chloroethoxy)methane	Water	20,2.9	85,15.0	1.2		3
	Sediment	20,2.9	85,15.0	42		3
Bis(2-chloroethyl)ether	Water	23,13.7	80,11.3	1.4		3
	Sediment	23,13.7	80,11.3	49		3
Bis(2-chloroisopropyl)ether	Water	28,6.0	93,5.2	2.1		3
	Sediment	28,6.0	93,5.2	74		3
Bis(2-ethylhexyl)phthalate	Water	21,5.1	73,8.3	2		3
	Sediment	21,5.1	73,8.3	70		3
4-Bromophenyl phenyl ether	Water	20,7.6	96,3.6	1.6		3
	Sediment	20,7.6	96,3.6	56		3
Butyl benzyl phthalate	Water	12,4.6	81,5.0	2.1		3
	Sediment	12,4.6	81,5.0	74		3
p-Chloraniline	Water			1.2		
	Sediment	20,6.1	120,15.0	42		3
4-Chloro-3-methylphenol (p-chloro-m-cresol)	Water	15,4.6	93,6.5	2.7		3
	Sediment	15,4.6	93,6.5	95		3
2-Chloronaphthalene	Water	21,6.1	91,6.0	1.1		3
	Sediment	21,6.1	91,6.0	39		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
2-Chlorophenol	Water	25,4.7	86,3.0	1.0		3
	Sediment	25,4.7	86,3.0	35		3
4-Chlorophenyl phenyl ether	Water	17,9.0	104,7.0	2.7		3
	Sediment	17,9.0	104,7.0	95		3
Chrysene	Water	21,4.6	95,19.0	1.9		3
	Sediment	21,4.6	95,19.0	67		3
2-Methylphenol (o-cresol)	Water			1.3		
	Sediment	20,4.5	114,4.6	1.3		3
4-Methylphenol (p-cresol)	Water			1.5		
	Sediment	21,9.3	112,11.0	1.5		3
Dibenzo(a,h)anthracene	Water	15,10.1	112,12.2	0.9		3
	Sediment	15,10.1	112,12.2	32		3
Dibenzofuran	Water	16,1.1	98,10.0	2.5		3
	Sediment	16,1.1	98,10.0	88		3
Di-n-butyl phthalate	Water	11,8.3	99,15.9	1.2		3
	Sediment	11,8.3	99,15.9	42		3
1,2-Dichlorobenzene	Water	12,7.0	89,6.0	1		3
	Sediment	12,7.0	89,6.0	35		3
1,3-Dichlorobenzene	Water	15,9.4	100,1.8	1.3		3
	Sediment	15,9.4	100,1.8	45		3
1,4-Dichlorobenzene	Water	15,2.8	91,11.6	1.2		3
	Sediment	15,2.8	91,11.6	42		3

**TABLE 3.1--Continued  
QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
3,3'-Dichlorobenzidine	Water	15,5.3	99,1.6	3.3		3
	Sediment	15,5.3	99,1.6	115		3
2,4-Dichlorophenol	Water	13,6.6	107,2.7	2.1		3
	Sediment	13,6.6	107,2.7	74		3
Diethyl phthalate	Water	10,5.4	96,4.3	2		3
	Sediment	10,5.4	96,4.3	70		3
2,4-Dimethylphenol	Water	10,5.4	99,1.7	2.7		3
	Sediment	10,5.4	99,1.7	95		3
Dimethyl phthalate	Water	12,5.1	100,4.4	1.6		3
	Sediment	12,5.1	100,4.4	56		3
4,6-Dinitro-2-methylphenol	Water	12,5.1	96,3.8	48		3
	Sediment	12,5.1	96,3.8	1,670		3
2,4-Dinitrophenol	Water	17,4.1	106,5.5	76		3
	Sediment	17,4.1	106,5.5	2,650		3
2,4-Dinitrotoluene	Water	12,2.6	114,1.1	13		3
	Sediment	12,2.6	114,1.1	450		3
2,6-Dinitrotoluene	Water	10,1.9	99,1.2	8.8		3
	Sediment	10,1.9	99,1.2	310		3
Di-n-octyl phthalate	Water	15,6.1	97,2.7	3.2		3
	Sediment	15,6.1	97,2.7	110		3
Fluoranthene	Water	12,1.7	110,9.2	0.8		3
	Sediment	12,1.7	110,9.2	28		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
Fluorene	Water	11,1.0	109,6.0	2.5		3
	Sediment	11,1.0	109,6.0	88		3
Hexachlorobenzene	Water	20,7.6	105,10.1	2.7		3
	Sediment	20,7.6	105,10.1	95		3
Hexachlorobutadiene	Water	18,7.3	103,16.6	1.7		3
	Sediment	18,7.3	103,16.6	60		3
Hexachlorocyclopentadiene	Water			8.8		
	Sediment	16,1.6	100,3.8	330		3
Hexachloroethane	Water	15,3.9	98,6.1	3.1		3
	Sediment	15,3.9	98,6.1	110		3
Indeno(1,2,3-cd)pyrene	Water	15,2.8	88,9.8	1.3		3
	Sediment	15,2.8	88,9.8	45		3
Isophorone	Water	15,9.4	100,1.8	1.5		3
	Sediment	15,9.4	100,1.8	53		3
2-Methylnaphthalene	Water			1		
	Sediment	13,6.6	107,2.7	35		3
Naphthalene	Water	16,3.9	104,2.4	0.6		3
	Sediment	16,3.9	104,2.4	21		3
o-Nitroaniline	Water			2.2		
	Sediment	15,5.4	96,11.0	77		3
m-Nitroaniline	Water			8.5		
	Sediment	15,7.6	96,7.0	300		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
p-Nitroaniline	Water			4.2		
	Sediment	16,1.1	98,10.0	150		3
Nitrobenzene	Water	13,2.6	97,5.6	1.5		3
	Sediment	13,2.6	97,5.6	53		3
2-Nitrophenol	Water	12,1.9	97,2.3	2.8		3
	Sediment	12,1.9	97,2.3	98		3
4-Nitrophenol	Water	12,3.0	93,11.7	32		3
	Sediment	12,3.0	93,11.7	1,130		3
N-Nitroso-di-phenylamine	Water	12,3.6	90,6.1	0.8		3
	Sediment	12,3.6	90,6.1	28		3
N-Nitroso-di-n-propylamine	Water	12,3.6	90,6.1	2		3
	Sediment	12,3.6	90,6.1	70		3
Pentachlorophenol	Water	22,5.3	96,5.2	39		3
	Sediment	22,5.3	96,5.2	1,350		3
Phenanthrene	Water	17,1.7	95,10.0	0.8		3
	Sediment	17,1.7	95,10.0	28		3
Phenol	Water	21,5.6	92,5.7	1.4		3
	Sediment	21,5.6	92,5.7	49		3
Pyrene	Water	30,8.9	62,16.0	2.4		3
	Sediment	30,8.9	62,16.0	84		3
1,2,4-Trichlorobenzene	Water	12,6.1	97,10.6	1.9		3
	Sediment	12,6.1	97,10.6	67		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
2,4,5-Trichlorophenol	Water			7		
	Sediment	11,3.2	93,7.0	245		3
2,4,6-Trichlorophenol	Water	13,9.1	91,10.7	11		3
	Sediment	13,9.1	91,10.7	370		3
Aldrin	Water	16,4	81,4	0.02		3
	Sediment	16,4	81,4	0.8		3
alpha-BHC	Water	13,4	84,3	0.02		3
	Sediment	13,4	84,3	0.8		3
beta-BHC	Water	22,2	81,7	0.02		3
	Sediment	22,2	81,7	0.8		3
gamma-BHC	Water	18,9	81,7	0.02		3
	Sediment	18,9	81,7	0.7		3
delta-BHC	Water	12,6	82,5	0.02		3
	Sediment	12,6	82,5	0.8		3
alpha Chlordane	Water	13,13	82,4	0.12		3
	Sediment	13,13	82,4	4.2		3
gamma Chlordane	Water			0.23		
	Sediment			8.0		
4-4' -DDD	Water	20,18	84,30	0.02		3
	Sediment	20,18	84,30	0.7		3
4-4' -DDE	Water	13,6	85,14	0.02		3
	Sediment	13,6	85,14	0.6		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

Component	Matrix	Precision x RPD, Sd	Accuracy % Rec, sd	MDL (ppb)	Method	Reference
4,4' -DDT	Water	17,39	93,13	0.02		3
	Sediment	17,39	93,13	0.7		3
Endosulfan I	Water	10,7	97,4	0.02		3
	Sediment	10,7	97,4	0.7		3
Endosulfan II	Water	41,65	93,34	0.03		3
	Sediment	41,65	93,34	1.1		3
Endosulfan sulfate	Water	13,33	89,37	0.02		3
	Sediment	13,33	89,37	0.8		3
Endrin	Water	20,25	89,4	0.04		3
	Sediment	20,25	89,4	1.4		3
Endrin ketone	Water			0.3		
	Sediment			0.9		
Heptachlor	Water	6,13	69,4	0.02		3
	Sediment	6,13	69,4	0.7		3
Heptachlor epoxide	Water	18,11	89,10	0.02		3
	Sediment	18,11	89,10	0.8		3
Methoxychlor	Water			22.4		
	Sediment			1.3		
Toxaphene	Water	9,32	80,17.4	2.5		3
	Sediment	9,32	80,17.4	65		3
PCB-1016	Water	13,1.5	81,50	1.0		3
	Sediment	13,1.5	81,50	33.3		3

**TABLE 3.1--Continued  
 QUALITY ASSURANCE OBJECTIVES**

<b>Component</b>	<b>Matrix</b>	<b>Precision x RPD, Sd</b>	<b>Accuracy % Rec, sd</b>	<b>MDL (ppb)</b>	<b>Method</b>	<b>Reference</b>
PCB-1221	Water	29,7.6	96,65	3.0		3
	Sediment	29,7.6	96,65	100		3
PCB-1232	Water	21,19.3	91,108	2.0		3
	Sediment	21,19.3	91,108	66.7		3
PCB-1242	Water	21,19.3	91,108	1.0		3
	Sediment	21,19.3	91,108	33.3		3
PCB-1248	Water	21,19.3	91,108	2.0		3
	Sediment	21,19.3	91,108	66.7		3
PCB-1254	Water	21,19.3	91,108	1.0		3
	Sediment	21,19.3	91,108	33.3		3
PCB-1260	Water	21,19.3	91,108	1.0		3
	Sediment	21,19.3	91,108	33.3		3

NA - Not Applicable  
 ID - Insufficient Data

- References:
1. EPA 600/4-79-020, Revised March 1983
  2. Standard Methods, 17th Edition, 1989
  3. CLP-SOW for Organics, February 1988, as revised through 1989
  4. CLP-SOW for Inorganics July 1988, as revised through 1989.

MDL (Method Detection Limits): Detection limits are determined by using 40 CFR, Part 136, Appendix B. Some detection limits are based upon ideal conditions representing the best data achievable for clean sample. The Detection Limits are dependent and may vary upon sample matrix and dilution.

## **SECTION 4 SAMPLING PROCEDURES**

The SAP details decontamination procedures and sample collection procedures and locations.

## SECTION 5 SAMPLE CUSTODY

Proper custody procedures are needed to ensure that samples have been obtained from the locations stated and that samples have reached the laboratory without alteration. All sample bottles shall be maintained in a locked storage area prior to use. Evidence of the sample traceability from collection to shipment, laboratory receipt, and laboratory custody must be documented. A sample is considered to be in a person's custody if the sample is:

- In a person's actual possession;
- In view after being in a person's possession;
- Locked so no one can tamper with it after having been in physical custody; or
- In a secured area, restricted to authorized personnel.

### 5.1 FIELD CHAIN-OF-CUSTODY PROCEDURES

Each person involved with data and/or sample handling shall be trained in chain-of-custody procedures prior to the implementation of the field program. To reduce the chance for error, the number of personnel handling the samples will be restricted. Sample handling, chain-of-custody, and sample designation and documentation procedures are discussed in Sections 5 and 7 of the Sampling and Analysis Plan.

### 5.2 LABORATORY CUSTODY PROCEDURES

All samples will be shipped to the laboratory accompanied by a chain-of-custody record, as described in Section 7 of the FSP. Immediately upon receipt, samples are logged into the computer that tabulates test assignments and analytical data. This program assures that all tests are performed in a timely manner, while assuring quality and completeness. After being logged in, the samples are placed in a designated, clean, dry room with refrigeration. Once the samples analyses are completed, the unused portion of the sample, together with identifying labels and other documentation, are returned to storage.

At the end of a project, all custody forms will be returned to the laboratory project manager and the original custody forms will be returned to ES. Copies of

custody information will be retained in the reporting laboratories' client files. Hard copies of reports, chain-of-custody forms and sample registries will be kept on file for a period of ten years following completion of the project.

### **5.3 DOCUMENT CONTROL**

#### **5.3.1 Maintenance of Records**

This project will require the administration of a central project file. The data and records management protocols will provide adequate controls and retention of all materials related to the project. Record control will include receipt from external sources, transmittals, transfer to storage and indication of record status. Record retention will include receipt at storage areas, indexing, filing, storage, maintenance, and retrieval.

#### **5.3.1 Record Control**

All incoming materials related to the project including sketches, correspondence, authorizations, and logs shall be forwarded to the project manager or designated assistant. These documents will be placed in the project file as soon as is practical. If correspondence is needed for reference by project personnel, a copy will be made rather than retaining the original. All records shall be legible and easily identifiable.

Examples of the types of records that will be maintained in the project file are:

- Field documents;
- Correspondence;
- Photographs;
- Laboratory data;
- Reports; and
- Procurement agreements.

Outgoing project correspondence and reports must be reviewed and signed by the project manager prior to mailing. The office copy of all outgoing documents shall bear distribution information.

Analytical reports will be transmitted from the laboratory to the ES QA Officer. Upon receipt, the analytical data will be stamped "Received" followed by the date of receipt. The original data will be maintained in the ES project file. Photocopies of the reports will be used for data reduction and validation.

### 5.3.2 Record Status

To prevent the inadvertent use of obsolete or superseded project-related procedures, all personnel of the laboratory and/or project staffs will be responsible for reporting changes in protocol to the project manager and/or the laboratory manager. The project manager and/or laboratory manager will then inform the project and laboratory staffs and the project quality assurance officer of these changes.

Revisions to procedures shall be subject to the same level of review and approval as the original document. The revised document will be distributed to all holders of the original document and discussed with project personnel. Outdated procedures will be marked "void". The voided document may be destroyed at the request of the project manager. However, one copy of the voided document will be maintained in the project file. The reasons for and the date the document was voided should be recorded.

### 5.3.3 Record Storage

All project-related information will be maintained by the Escambia County Solid Waste Department. Designated personnel will assure that incoming records are legible and are in suitable condition for storage. A records index will be initiated at the beginning of the overall project. Each document that is placed into the project file will be logged. The logging of the records will be the responsibility of the project manager and/or his designee.

Record storage will be performed in two stages:

- Storage during and immediately following the project; and
- Long-term storage of records directly related to the project.

Both phases will use storage facilities that provide a suitable environment to minimize deterioration or damage and that prevents loss. The facilities will, where possible, have controlled access and will provide protection from excess moisture and temperature extremes. Records will be secured in steel file cabinets labeled with the appropriate project identification.

The removal of records from all files during both stages will be controlled by the use of withdrawal cards.

At the completion of the project, the project manager or his appointed document custodian will be responsible for inventorying the project file. The records contained in the project file will be compared against the records listed on the file index sheets; discrepancies must be resolved prior to transferring the file to a permanent storage facility. All material from the project file, including drawings,

project-related quality assurance documents, and software program documentation and verification records will be permanently stored. Duplicate records may be made and stored at a separate location.

All storage systems will provide for the prompt retrieval of information for reference or use outside the storage areas. Project records will be accessible for a period of three years after close of this project.

#### **5.3.4 On-Site Control**

A file, similar to the project central file, will be established and maintained by the field personnel under the direction of the field team leader. Upon completion of the field program, the on-site file will be transferred to, and integrated with, the office project files.

## SECTION 6 CALIBRATION PROCEDURES AND FREQUENCY

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

### 6.1 FIELD EQUIPMENT

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments will include a pH and temperature meter, specific conductivity meter, photoionization detector, and PM<sub>10</sub> and TSP/Metals samplers. The calibration of each field instrument will be documented on the calibration log.

The pH meter will be calibrated with standard buffer solutions prior to a field trip. In the field, the meter will be calibrated daily with two buffers before use. Thereafter, the meter will be checked against two buffers as deemed necessary by the Field Team Leader. Fresh traceable buffer solutions will be used for each field trip. Calibration procedures and frequency will be recorded in a field log book, along with the lot numbers of the buffers.

The specific conductivity meter will have its conductivity cells cleaned and checked against known conductivity standards prior to the field effort. In the field, the instrument will be checked daily with traceable standards.

Both PM<sub>10</sub> and TSP/Metals samplers will be equipped with Accu-vol flow controllers and are calibrated according to the same procedure. The procedure is specified in 40 CFR 50, Appendix B, and is discussed in the SAP, Appendix B. Calibration of the sampling equipment for volatile emissions is discussed in Appendix C of the SAP.

### 6.2 LABORATORY EQUIPMENT

Calibration of laboratory equipment will be conducted in accordance with Exhibit D of the 1988 CLP SOW (as revised through 1989). Records of calibration, repairs, or replacement will be filed and maintained by the designated laboratory personnel performing quality control activities. These records shall be filed at the location where the work is performed and will be subject to QA audit. For all

instruments, the laboratory shall maintain a factory-trained repair staff with in-house spare parts or shall maintain service contracts with vendors. Specific Kiber Analytical Services, Inc. procedures have been provided in Appendix B for several analyses.

## SECTION 7 ANALYTICAL PROCEDURES

Analytical procedures will be conducted in accordance with standard procedures, EPA methods, and generally accepted protocols. This section identifies the analytical procedures to be used by the laboratory. Modifications, where necessary to achieve the DQOs, are appropriately noted. A summary of the analytical program is presented in Table 7.1. Procedures used by Kiber Analytical Services for general GC/MS, GC/MS volatiles analysis, and GC/MS semivolatiles analysis are provided in Appendix B.

### 7.1 CHEMICAL ANALYSES

#### 7.1.1 CLP Methods

Analysis of samples for VOCs, semivolatile organics, and pesticides/PCBs will be conducted in accordance with the February 1988 CLP SOW for Organics Analysis (as revised through 1989), Multi-Media, Multi-Concentration. Analysis of samples for inorganics will be conducted in accordance with the July 1988 CLP SOW for Inorganics Analysis (as revised through 1989), Multi-Media, Multi-Concentration. References to specific exhibits and paragraphs in the CLP SOW refer to the exhibit or section in both CLP SOWs, unless otherwise stated.

All analyses will be performed as specified in Exhibit D of the CLP SOW. Exceptions to the Exhibit D procedures are noted below:

- Section II, Holding times - all holding times will be calculated from the date of sample collection, as identified on the chain-of-custody records.

#### 7.1.2 Non-CLP Methods

The ions chloride, fluoride, sulfate, nitrate, and nitrite will be analyzed by EPA Methods 9250, 340.2, 9038, 352.1, and 354.1, respectively. Analysis of volatile components in air samples will be by EPA Method TO14-1.

## 7.2 PHYSICAL PARAMETERS

The following parameters will be evaluated for water samples:

- Total dissolved solids (EPA 160.1);
- Total organic carbon (EPA 9060);
- Conductivity (EPA 120.1);
- pH (EPA 9040);
- Temperature (EPA 170.1);
- Dissolved oxygen (EPA 360.1); and
- Turbidity (EPA 180.1).

Testing for these parameters will be conducted in accordance with the indicated non-CLP method.

**TABLE 7.1**  
**SUMMARY OF SAMPLING AND ANALYSIS PROGRAM**

<b>Analysis</b>	<b>Proposed Analytical Method<sup>(1)</sup></b>	<b>Maximum No. of Samples<sup>(2)</sup></b>
<b>Soils and Sediment</b>		
TCL BNA Extractables	CLP-SOW	35
TCL Volatile Organics	CLP-SOW	35
TCL Pesticides/PCBs	CLP-SOW	35
TAL Metals	CLP-SOW	35
<b>Groundwater</b>		
TCL BNA Extractables	CLP-SOW	21
TCL Volatile Organics	CLP-SOW	27
TCL Pesticides/PCBs	CLP-SOW	21
TAL Metals	CLP-SOW	21
Other Inorganic Targets	EPA Methods	21
<b>Surface Water</b>		
TCL BNA Extractables	CLP-SOW	8
TCL Volatile Organics	CLP-SOW	8
TCL Pesticides/PCBs	CLP-SOW	8
TAL Metals	CLP-SOW	8
Other Inorganic Targets	EPA Methods	8
<b>Ambient Air</b>		
Respirable particulate matter	40 CFR 50, Apps. B, G, and J	5
TCL/TAL Scan	CLP-SOW	5
Volatile Organics	EPA TO14-1	5

(1)Contract Laboratory Program, Statement of Work 1988 (including revisions 1989).

(2)Sample quantities include QA/QC samples. Trip, rinsate, and field blanks have been included in the groundwater totals.

## SECTION 8 INTERNAL QUALITY CONTROL CHECKS

### 8.1 FIELD QUALITY CONTROL CHECKS

As a check on field QA/QC, trip blanks, field blanks, equipment rinseate samples and field duplicates will be sent to the laboratory with specified frequencies. The frequency with which these samples will be taken is discussed in Section 3.

### 8.2 LABORATORY QUALITY CONTROL CHECKS

Quality control data are necessary to determine precision and accuracy of the analyses and to demonstrate the absence of interferences and contamination of glassware and reagents. A copy of the Kiber Analytical Services, Inc. Quality Assurance/Quality Control Program is provided in Appendix A.

#### 8.2.1 VOC QA/QC Requirements

The minimum QC requirements for VOC analyses are specified in Exhibit E, Section IV-VOA, of the February 1988 CLP SOW (as revised through 1989). For organics analysis the required QA/QC operations include:

- GC/MS Mass Calibration and Ion Abundance Patterns;
- GC/MS Initial and Continuing Calibration;
- Stability of Internal Standard Responses and Retention Times;
- Method Blank Analysis;
- System Monitoring Compound Recoveries;
- Matrix Spike and Matrix Spike Duplicate Analyses; and
- Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates.

### **8.2.2 Semivolatile QA/QC Requirements**

The minimum QC requirements for semivolatile analyses are specified in Exhibit E of the February 1988 CLP SOW (as revised through 1989) for Organics Analyses. The required QA/QC operations include:

- GC/MS Mass Calibration and Ion Abundance Patterns;
- GC/MS Initial and Continuing Calibration;
- Stability of Internal Standard Responses and Retention Times;
- Method Blank Analysis;
- Surrogate Recoveries;
- Matrix Spike and Matrix Spike Duplicate Analyses; and
- Dilution of Samples, Matrix Spikes, and Matrix Spike Duplicates.

### **8.2.3 Pesticides/PCBs QA/QC Requirements**

The minimum QA requirements for pesticides/PCBs are specified in Exhibit E of the February 1988 CLP SOW (as revised through 1989) for organics analyses. The required QA/QC operations include:

- GC Column Resolution
- GC/EC Initial and Continuing Calibration
- Determination of Retention Times and Retention Time Windows
- Analytical Sequence
- Blank Analyses
- Surrogate Recoveries
- Matrix Spike and Matrix Spike Duplicate Analyses
- Dilution of Samples, Matrix Spike and Matrix Spike Duplicates

### **8.2.4 Inorganics QA/QC Requirements**

The minimum QC requirements for inorganics analyses are specified in Exhibit E of the July 1988 CLP SOW for Inorganics Analysis. The required QA/QC operations include:

- Instrument Calibration
- Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV)
- CRDL Standards for AA (CRA) and ICP (CRI);

- Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses;
- ICP Interference Check Sample (ICS) Analyses;
- Spike Sample Analysis (S)
- Duplicate Sample Analysis (D);
- Laboratory Control Sample (LCS) Analysis;
- ICP Serial Dilution Analysis (L);
- Instrument Detection Limit (IDL) Determination;
- Interelement Corrections for ICP (ICP);
- Linear Range Analysis (LRA); and
- Furnace AA QC Analyses.

## SECTION 9 DATA REDUCTION, VALIDATION, AND REPORTING

### 9.1 DATA REDUCTION

#### 9.1.1 Field Data

Field measurements will be made by competent field geologists, engineers, environmental scientists, and/or technicians.

Field data will be validated using four different procedures:

- Routine checks will be made during the processing of data. An example is looking for errors in identification codes.
- Internal consistency of a data set will be evaluated. This step will involve plotting the data and testing for outliers.
- Checks for consistency of the data set over time will be performed. This can be accomplished by visually comparing data sets against gross upper limits obtained from historical data sets, or by testing for historical consistency. Anomalous data will be identified.
- Checks may be made for consistency with parallel data sets, that is, data sets obtained presumably from the same population (for example, from the same region of the aquifer or volume of soil).

The purpose of these validation checks and tests is to identify outliers; that is, an observation that does not conform to the pattern established by other observations. Outliers may be the result of transcription errors or instrumental breakdowns. Outliers may also be manifestations of a greater degree of spatial or temporal variability than expected.

After an outlier has been identified, a decision concerning its fate must be rendered. Obvious mistakes in data will be corrected when possible, and the correct value will be inserted. If the correct value cannot be obtained, the data may be excluded. An attempt will be made to explain the existence of the outlier. If no plausible explanation can be found for the outlier, it may be excluded, but a note to that effect will be included in the report. Also, an attempt will be made to determine the effect of the outlier when both included and excluded in the data set.

### 9.1.2 Laboratory Data

Data reduction procedures for chemical analyses are specified in the CLP SOWs. Data reduction procedures for physical parameters are identified in the appropriate method. The laboratory will follow the specified procedures.

## 9.2 DATA REPORTING

Sample data packages will be prepared as specified in Exhibit B, Section II, Paragraph E of the February 1988 CLP SOW for Organics Analysis, and in Exhibit B, Section II, Paragraph D, of the July 1988 CLP SOW for Inorganics Analysis.

The data will also be supplied in computer-readable format on diskettes. The data will be formatted as specified in Exhibit B, Section II, Paragraph F, of the Organics CLP SOW, and in Exhibit B, Section 2, Paragraph E of the Inorganics CLP SOW, and in all exhibits referenced therein.

## 9.3 DATA REVIEW AND VALIDATION

The organic data packages received from the analytical laboratory will be reviewed and validated based on the EPA guidance document *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses* (EPA 1988c). The general criteria used to evaluate the laboratory's organic analytical performance will be based on:

- Data Completeness;
- Holding Times;
- GC/MS Tuning;
- Calibrations;
- Blanks;
- Surrogate Recoveries;
- Matrix Spike/Matrix Spike Duplicate;
- Field Duplicates;
- Internal Standard Performance;
- Instrument Performance;
- Compound Identification; and
- Compound Quantitation.

The general criteria used to evaluate the laboratory's inorganic analytical performance will be based on:

- Holding Times;
- Calibration Verification;
- Field and Lab Blank Analysis;
- Interference QC Results;
- Matrix Spike Percent Recovery Results;
- Laboratory Precision Evaluation;
- Field Precision Evaluation;
- Lab Control Sample Results;
- Detection Limit Results;
- Standard Addition Results; and
- Serial Dilution Results.

**TABLE 9.1  
 REQUIRED LABORATORY DELIVERABLES**

<b>Method Requirements</b>	<b>Laboratory Deliverables</b>
<b>Requirements for all methods:</b>	
- Project identification	Case narrative
- Field sample number	Signed chain-of-custody (COC) forms
- Laboratory sample number	Signed COC forms
- Sample matrix description	Signed COC forms
- Date of sample collection	Signed COC forms
- Date of sample receipt at laboratory	Signed COC forms
- Analytical method description and reference citation	Case narrative
- Dates of sample preparation and analysis (including first run and subsequent runs)	Specific deliverable depends upon type of analysis (see below)
- Quantitation limits achieved	Specific deliverable upon type of analysis (see below)
- Dilution or concentration factors	Specific deliverable depends upon type of analysis (see below)
<b>Requirements for all methods:</b>	
- Discussion of unusual circumstances or problems	Case narrative
- LCS with results on Quality Control Charts. Run with each batch of samples processed	Control charts
<b>Requirements for organic analytical methods:</b>	
- Sample data sheets	CLP Form 1 or equivalent
- GC/MS sample raw data	Gas chromatograms, mass spectra of individual GC peaks, and listings of retention time, quantitation ions, and peak areas

**TABLE 9.1--Continued  
 REQUIRED LABORATORY DELIVERABLES**

<b>Method Requirements</b>	<b>Laboratory Deliverables</b>
- GC and HPLC sample raw data	Chromatograms, and listings of retention time and peak areas.
- Surrogate recoveries. Surrogates to be used in volatiles, semi-volatiles, and pesticide/PCB analyses.	CLP Form 2 or equivalent and raw data
- Matrix spike/matrix spike duplicate	CLP Form 3 or equivalent and raw data.
- Method blank analysis	CLP Form 4 or equivalent and raw data.
<b>Requirements for organic analytical methods:</b>	
- GC/MS instrument performance check. Tuning and mass calibration using bromofluorobenzene (BFB) for method SW8240 and decafluoro-triphenylphosphine (DFTPP) for methods EPA 625 and SW8270.	CLP Form 5 or equivalent and raw data
- GC/MS initial calibration data for volatile and semivolatile analyses	CLP Form 6 or equivalent and raw data.
- Calibration data for pesticide/PCB analyses.	SW846 Form 9 or equivalent and raw data.
- GC initial calibration data for volatile and semivolatile analyses	
• if calibration factors are used	A form with five columns for multilevel calibration factors, and raw data
• if a calibration curve is used	A plot of the calibration curve is required with a linear regression determination (with flagged correlation coefficient if it is less than 0.995). Should also include raw data.
<b>Requirements for organic analytical methods:</b>	
- GC/MS continuing calibration data. No chromatograms or mass spectra are presented for calibration. These data should be filed in the laboratory and should be available if problems arise in reviewing/validating the data.	CLP Form 7 or equivalent

**TABLE 9.1--Continued  
 REQUIRED LABORATORY DELIVERABLES**

Method Requirements	Laboratory Deliverables
<p>The calibration information should be available for checking during on-site audits</p>	
<ul style="list-style-type: none"> <li>- GC continuing calibration data for volatile and semivolatile analyses. If calibration factors are used, calibration factors and their percent differences from the initial calibration must be reported. Retention time windows and analyte retention times must be included in this form.</li> </ul>	<p>A form similar to CLP Form 7, or equivalent.</p>
<ul style="list-style-type: none"> <li>- GC/MS internal standard area and retention time summary data</li> </ul>	<p>CLP Form 8 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- GC second column confirmation. To be done for all compounds that are detected above reporting limits.</li> </ul>	<p>Chromatograms of all confirmations of all samples and CLP Form 10 or equivalent for all positive results.</p>
<p><b>Requirements for inorganic analytical methods (metals and cyanide):</b></p>	
<ul style="list-style-type: none"> <li>- Sample data sheets</li> </ul>	<p>CLP Form 1 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Initial and continuing calibration</li> </ul>	<p>CLP Form 2 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Method blank, taken through sample preparation</li> </ul>	<p>CLP Form 3 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- ICP interference check sample</li> </ul>	<p>CLP Form 4 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Spike sample recovery</li> </ul>	<p>CLP Form 5A or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Post-digestion spike sample recovery for ICP metals</li> </ul>	<p>CLP Form 5B or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Post-digestion spike for GFAA</li> </ul>	<p>Recovery will be noted on raw data.</p>
<ul style="list-style-type: none"> <li>- Duplicate samples</li> </ul>	<p>CLP Form 6 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Laboratory control sample</li> </ul>	<p>CLP Form 7 or equivalent and raw data.</p>
<ul style="list-style-type: none"> <li>- Standard addition results</li> </ul>	<p>CLP Form 8 or equivalent and raw data.</p>

**TABLE 9.1--Continued  
 REQUIRED LABORATORY DELIVERABLES**

<b>Method Requirements</b>	<b>Laboratory Deliverables</b>
- ICP serial dilutions	CLP Form 9 or equivalent and raw data.
- Instrument detection limits (quarterly)	CLP Form 10 or equivalent.
<b>Requirements for inorganic analytical methods (metals and cyanide):</b>	
- ICP interelement correction factors (annually)	CLP Form 11A and 11B or equivalent.
- ICP linear ranges (quarterly)	CLP Form 12 or equivalent.
- Preparation log	CLP Form 13 or equivalent.
- Analysis run log	CLP Form 14 or equivalent.
<b>Requirements for other methods:</b>	
- Preparation and analysis logs	No format.
- Sample results	No format.
- Laboratory control sample	Control chart.
- Method blank results	No format.
- Initial Calibration results	No format.
- Continuing Calibration check	No format. Report percent relative standard deviation or percent difference from initial calibration.
- Spike/spike duplicate results	No format.

Note: CLP = Contract Laboratory Program, USEPA; PCP = polychlorinated biphenyls; GC = gas chromatography; MS = mass spectrometry; ICP = inductively - coupled plasma

## **SECTION 10 PERFORMANCE AND SYSTEM AUDITS**

### **10.1 EXTERNAL AUDITS**

Auditing of laboratories used in this project is the responsibility of EPA Region IV.

### **10.2 INTERNAL AUDITS**

Quality assurance audits will be performed by the ES Quality Assurance Officer (QAO) or designated alternate. The QAO will plan, schedule, and approve system and performance audits. These audits will be implemented to evaluate the capability and performance of project and subcontractor personnel, items, activities, and documentation of the measurement systems. At times, the QAO may request additional personnel with specific expertise from other project groups to assist in conducting performance audits. However, these personnel will not have participated in nor have responsibility for the direct work associated with the performance audit.

### **10.3 SYSTEM AUDITS**

System audits, performed by the QAO or his designee, will encompass evaluation of QA components to ascertain their appropriate selection and application. In addition, field and laboratory quality control procedures and associated documentation will be system audited. These audits will be performed once during the project; however, if conditions adverse to quality are detected, or if the Project Manager requests the QAO to perform unscheduled audits, these activities will be instituted.

### **10.4 PERFORMANCE AUDITS**

Performance audits will be conducted periodically through the life of the project to determine the accuracy and implementation of the Laboratory QA Plan. As in system audits, the QAO or assigned alternate will exercise planned and scheduled performance audits with the understanding that unplanned audits may be implemented if requested. Performance audits will be performed once the project is generating data.

## **SECTION 11 PREVENTIVE MAINTENANCE**

### **11.1 PREVENTIVE MAINTENANCE PROCEDURES**

Equipment, instruments, tools, gauges, and other items requiring preventive maintenance will be serviced in accordance with the manufacturer's specified recommendations and written procedure developed by the operators.

### **11.2 SCHEDULES**

Manufacturer's procedures identify the schedule for servicing critical items in order to minimize the downtime of the measurement system. It will be the responsibility of the operator to adhere to this maintenance schedule and to arrange any necessary and prompt service as required. Service to the equipment, instruments, tools, gauges, etc. shall be performed by qualified personnel.

In the absence of any manufacturer's recommended maintenance criteria, a maintenance procedure will be developed by the operator based upon experience and previous use of the equipment.

### **11.3 METHOD-SPECIFIED PREVENTIVE MAINTENANCE**

In the event that a CLP method mandates specific preventive maintenance procedures which are more frequent than that recommended by the manufacturer, then the frequency specified in the method shall be followed.

### **11.4 RECORDS**

Logs shall be established to record maintenance and service procedures and schedules. All maintenance records will be documented and traceable to the specific equipment, instruments, tools, and gauges.

Records produced for laboratory instruments shall be reviewed, maintained, and filed by the operators at the laboratories and by field personnel for equipment, instruments, tools, and gauges which are used at the site. The ES QAO will audit these records to verify complete adherence to these procedures.

## SECTION 12 DATA ASSESSMENT PROCEDURES

Procedures used to assess data precision and accuracy are in accordance with 44 FR 69533 "Guidelines Establishing Test Procedures for the Analyses of Pollutants," Appendix III (Example Quality Assurance and Quality Control Procedures for Organic Priority Pollutants), December 3, 1979. Completeness is recorded by comparing the number of parameters initially analyzed for with the number of parameters successfully completed and validated. For this project a target control limit of greater than 90 percent will be used for non-critical samples, and a limit of 100 percent will be used for critical samples.

### 12.1 ACCURACY

The percent recovery (PR) is calculated as below:

$$PR = \frac{S_s - S_o}{S_A} \times 100$$

$S_o$  = The background value, value obtained by analyzing the sample.

$S_A$  = Concentration of the spike added to the sample.

$S_s$  = Value obtained by analyzing the sample with the spike added.

% = Percent Recovery

### 12.2 PRECISION

The relative percent difference (RPD) is calculated as below:

$$RPD = \frac{V_1 - V_2}{(V_1 + V_2)/2} \times 100$$

$V_1, V_2$  = The 2 values obtained by analyzing the duplicate samples.

### 12.3 COMPLETENESS

The percent complete (PC) is calculated as follows:

$$PC = \frac{N_A}{N_I} \times 100$$

$N_A$  = Actual number of valid analytical results obtained

$N_I$  = Theoretical number of results obtainable under ideal conditions.

## **SECTION 13 CORRECTIVE ACTIONS**

The following procedures have been established to assure that conditions adverse to quality including malfunctions, deficiencies, deviations, and errors are promptly investigated, documented, evaluated, and corrected.

### **13.1 INITIATION AND APPROVAL OF CORRECTIVE ACTION**

When a significant condition adverse to quality is noted at the project site, laboratory, or subcontractor locations, the cause of the condition will be determined and corrective action taken to preclude repetition. Condition identification, cause, reference documents, and corrective action planned to be taken will be documented and reported to the Project Manager, Quality Assurance Officer (QAO), Field Team Leader, and involved subcontractor management, as a minimum. Implementation of correction action will be verified by documented follow-up action. All project personnel have the responsibility, as part of their normal work duties, to promptly identify, solicit approved correction, and report conditions adverse to quality.

Corrective actions may be initiated as a minimum:

- When predetermined acceptance standards are not attained (objectives for precision, accuracy and completeness);
- When procedures or data compiled are determined to be faulty;
- When equipment or instrumentation is found faulty;
- When samples and test results cannot be traced with certainty;
- When quality assurance requirements have been violated;
- When designated approvals have been circumvented;
- As a result of system and performance audits;
- As a result of a management assessment; or
- As a result of laboratory/inter-laboratory comparison studies.

### **13.2 PROCEDURE DESCRIPTION**

Project management and staff including field investigation teams, quality assurance auditors, document and sample control personnel, and laboratory groups monitor ongoing work performance in the normal course of daily responsibilities. Work will be audited at the site, laboratories, and subcontractor locations by the QAO or his designee and/or by QA personnel of the state of Florida or EPA. Items, activities, or documents ascertained to be in noncompliance with quality assurance requirements will be documented and corrective actions mandated through the audit report. Corrective actions are logged, maintained, and controlled by the QAO.

Following identification of an adverse condition or quality assurance problem, notification of the deficiency will be made to the Project Manager and the senior individual in charge of the activity found to be deficient along with recommendations for correction. A record of this notification will be attached to the audit report. Following implementation of corrective action, the senior individual in charge will report actions taken and results to the Project Manager and QAO. The QAO will notify the Project Manager when conditions adverse to quality have been corrected. A record of action taken and results will also be attached to the audit report.

### **13.3 FIELD CHANGES**

Any deviation from project requirements as specified in this document requires proper documentation using a Work Plan Modification form. This form will be completed in the field by the Field Team Leader and forwarded to the EPA RPM and to the EPA Project Manager by the most expedient communications means available. Upon receipt, the RPM and PM will review and indicate final disposition of the request and return the original document to the originator. A copy of the document should be retained for the project file. Changes that require an immediate response will be initiated by telephone and then documented using the procedure described above.

## **SECTION 14 QUALITY ASSURANCE REPORTS**

Quality assurance reports to management consist of the reports on audits, reports on correction of deficiencies found in audits, a final QA report on field sampling activities, and a final analytical laboratory QA/QC report.

### **14.1 FIELD QA AUDITS**

The mobilization stage will be audited before work begins to assure that all procedures, training, and materials are ready to support the QA plan.

Field activities may be audited during operation in order to assure compliance with the QA plan. Additional audits may be required depending on the results of these audits.

All audits and corrective action(s) will be reported in writing to the Program Manager and the Project Manager.

### **14.2 QUALITY ASSURANCE REPORTS TO MANAGEMENT, CLIENTS, AND AGENCIES**

On a monthly basis while analyses are being performed, the laboratory QA Manager will review all QC notebooks, control criteria, and perform a system audit. Section QC coordinators will review the notebooks, control criteria and all QA nonconformance/corrective action reports on a monthly basis. The purpose of this review is to determine if corrective action has been implemented. Each notebook will contain a QA Surveillance Report which is signed during each review.

Non-implementations of corrective action will be listed on the QA surveillance reports with deadlines for implementation. QA surveillance reports with non-implementation will be filed by the QA Manager in a working file. These reports will not be signed and filed as complete by QC coordinators or QA managers until all corrective actions are implemented.

Each month samples are in process, the laboratory QA manager will prepare a Laboratory QA Report for the laboratory director and for ES.

The Monthly Laboratory QA Report will contain:

1. Copies of typed monthly QA surveillance reports (system audits) for each department;
2. Copies of all systems audit reports by external or internal auditors and all corrective actions and responses to these audits;
3. Results for all performance audits on external or internal samples including corrective actions/responses to these audits;
4. A list by department of all significant QA problems with recommended solutions;
5. A list by department of all performance and system audit failures which have not been addressed with recommended action; and
6. Suggested updates for the QA manual including control limits.

## SECTION 15 REFERENCES

- EPA, 1980, *Interim Guidelines and Specifications for Preparing Quality Assurance Project Plan*, EPA/QAMS-005/80, U.S. Environmental Protection Agency, Washington, D.C.
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**APPENDIX A**  
**KIBER ANALYTICAL SERVICES, INC. - QA/QC PROGRAM**

**QUALITY ASSURANCE /  
QUALITY CONTROL PROGRAM**

**KIBER ASSOCIATES, INC.  
KIBER ANALYTICAL SERVICES, INC.**

**ANALYTICAL AND TREATABILITY STUDY SERVICES  
ENVIRONMENTAL SPECIALISTS**

**4000 DEKALB TECHNOLOGY PARKWAY, N.E.  
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**SEPTEMBER 1990**

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FOR KIBER ASSOCIATES, INC./KIBER ANALYTICAL

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## I. PLAN DESCRIPTION

The primary objective of the Quality Assurance/Quality Control (QA/QC) plan is to assure that Kiber Associates, Inc./Kiber Analytical Services, Inc. (Kiber) generates scientifically valid, reproducible data for all treatability and analytical procedures. This is accomplished by meeting five criteria; Accuracy, Comparability, Precision, Representativeness, and Completeness.

The purpose of this QA/QC plan is to a) to define the nature of quality assurance for all analyses, b) to set forth the requirements for preparation of the QA procedures; and c) to describe the methods and procedures used in carrying out the QA/QC program.

Kiber's QA/QC Program contains component Sampling, Treatability Study, and Analytical Quality Control programs. The Sampling Program outlines sampling procedures, preservation, shipping, and Chain-of-Custody requirements. The Treatability Study Program combines treatment procedures, blending techniques, and analytical and physical properties considerations with knowledge of full scale operations. The Analytical QC Program delineates a system of standard procedures employed by all laboratory personnel for sample analysis as well as documentation.

The following document summarizes the organization of Kiber Associates and Kiber Analytical Services QA/QC Program from initial sampling through generation of the final report.

## II. ORGANIZATIONAL STRUCTURE

### Project Manager

For each project awarded to Kiber, a qualified Project Manager will be assigned, whether that project is a sampling effort, a treatability study, or analytical evaluation. The Project Manager is responsible for:

- A. Scheduling the work and personnel assigned to that project.
- B. Monitoring the progress of the work and responding to client inquiries.
- C. Verifying that activities are well documented and coordinated with the client's expectations.
- D. Verifying that all procedures are correct and adequate for the intended purpose.
- E. Confirming that the procedures conform to the client's specifications with respect to methods and frequency of sampling and analysis.
- F. Ensure conformity with any applicable regulatory criteria.

### Technical Director

The Technical Director at Kiber is primarily tasked with ensuring the management of all treatability services. Since procedures governing treatability studies are not published as with sampling and analysis, Kiber has developed specific procedures to be followed for each individual treatability study. The Technical Director is tasked with the following:

- A. Coordinating treatability procedures with the client to determine the specific scope of work.
- B. Offering suggestions to the client to ensure completeness of the study and results adequate for the intended use.

- C. Assigning a Project Manager to the study who is familiar with the type and scope of the project.
- D. Overall direction of the project.

#### Laboratory Director

The Laboratory Director is responsible for the overall operation of the laboratory. His responsibilities are as follows:

- A. Overall administration of laboratory activities.
- B. Direction of QA/QC Program
- C. Hiring, assignment and review of personnel
- D. Evaluation and purchase of equipment and instrumentation.
- E. Training and professional development.
- F. Review of proposals, bids, and quotations.

#### Quality Control Officer

The QC Officer reports directly to the Laboratory Director. The QC Officer has sufficient authority and organizational flexibility to identify problems, to initiate solutions, and to verify their implementation. The QC Officer's responsibilities are as follows:

- A. Develop QA procedures;
- B. Maintain surveillance over all routine and non-routine aspects of the QA program;
- C. Evaluate QC data reports;
- D. Performing inspections;

- E. Coordination of QA activities (e.g. propose changes of policy or procedures) among Directors, Managers and Supervisors;
- F. Managing interlaboratory comparison programs to assure full and prompt participation, evaluation of results and of all benefits relating therefrom;

#### Department Supervisors

Supervisors are assigned to each analytical division. Their responsibilities include:

- A. Assistance in preparation of QA plans and procedures for their individual departments.
- B. Ensuring that all personnel within their division are aware of relevant QA information;
- C. Informing the QC Officer of any deficiencies or situations requiring immediate attention relating to QA;
- D. Assuring that personnel are provided with continuing education or training, as necessary, for proper performance of the analytical work;
- E. Verifying that records are completed and maintained for each analysis performed in their department;
- F. Maintaining and calibrating equipment to the extent and at the frequency necessary to ensure accurate results;

#### Senior Chemist

The Senior Chemist is responsible for:

- A. Development and review of QA procedures;
- B. Making recommendations for technical decisions;
- C. Evaluating and reviewing test procedures;

- D. Ensuring that approved analytical procedures are used when possible or that state-of-the-art methods are used when approved methods are not available;
- E. Issuing stop-work orders for work which is not in compliance with requirements;
- F. Directing laboratory certification programs;
- G. Reviewing and signing laboratory reports;

#### Administrative Officer

The Administrative Officer is responsible for:

- A. General office operations
- B. Computer operations
- C. Scheduling and expediting
- D. Shipping and receiving;
- E. Chain-of-Custody and sample security;
- F. Sample Management;
- G. Report and document generation;
- H. Purchasing and inventory control;
- I. Client billing.

#### Technicians

Technicians are responsible for:

- A. Performing sample preparations and analyses;
- B. Maintaining a clean working environment;
- C. Performing QA/QC procedures in accordance with laboratory policy and recording all data;

### III. PERSONNEL QUALIFICATIONS

#### GENERAL

All persons hired or assigned to the positions described in this document receive on the job training by working with experienced personnel. Their work is closely supervised and evaluated. The principal criterion for employment or assignment is demonstrated professional proficiency at Kiber.

#### Laboratory Staff Members

We have developed an organization of technical specialists in all major disciplines of the environmental sciences. Each person is thoroughly trained and experienced in his/her respective field and qualified to function with other staff members to form an integrated team.

Required educational and experience qualifications of Laboratory personnel are described below:

Laboratory Director: Minimum B.S. Degree in Chemistry from an ACS accredited school, with minimum of 10 years experience.

Technical Director: Minimum B.S. Degree in environmental related field, with a minimum of 8 years experience.

Senior Chemist: Minimum M.S. Degree in Chemistry from an ACS accredited school, with a minimum of 5 years experience.

Quality Assurance Officer: Minimum B.S. Degree in Chemistry from an ACS accredited school, with a minimum of 5 years experience.

Section Chiefs Spectroscopy, Chromatography: Minimum B.S. Degree in Chemistry from an ACS accredited school with a minimum of 5 years experience. Directs, plans and performs analytical effort within the section.

Chemists: Minimum B.S. Degree in Chemistry from an ACS accredited school, with a minimum of 2 years experience.

Technicians: B.S. or B.A. Degree in Scientific Discipline or equivalent laboratory experience.

## IV. RECORDS

### GENERAL

Records are maintained which include the processing of all steps of a client's order, from the specification of the order to the delivery of the results. These records detail the sample identification, the date of performance, the analytical procedures followed, the results obtained, the measure of precision and accuracy achieved and the identities of those who performed the analyses, and reviewed and approved the results.

### Specific Records Maintained

Records providing information in the following areas are maintained either in laboratory notebooks, files or computer storage:

- a. Statements of Laboratory Policy
- b. Analytical Procedures
- c. Client's orders, proposals and contracts
- d. Sample collection
- e. Sample receipts
- f. Sample description and laboratory identification number
- g. Sample preparation
- h. Analytical results
- i. Reviews and approvals
- j. Reports submitted to clients
- k. Precision checks of instruments
- l. Results of analyses of quality control samples
- m. Results of interlaboratory analyses
- n. Verifications of computer and personnel computations
- o. Quality assurance reports
- p. Preparation of standards and carrier solutions

### Period of Record Retention

Statements of policies and procedures are signed and dated by the Laboratory Director. Superseded procedures are retained permanently on file. All records are maintained on computer disks.

## V. CHAIN-OF-CUSTODY PROCEDURES

Sample chain-of-custody is maintained when required by the client or regulatory agency. All samples that are collected by Kiber personnel are maintained under chain-of-custody control. This procedure insures that samples are collected, transferred, stored, analyzed and disposed of only by authorized personnel.

Immediately upon receipt, samples are logged into the computer which tabulates test assignments and analytical data. This program assures that all tests are performed in a timely manner, while assuring quality and completeness. After being logged in, the samples are placed in a designated, clean, dry room with refrigeration. Once the samples analyses are completed, the unused portion of the sample, together with identifying labels and other documentation are returned to storage.

## VI. QUALITY CONTROL IN SAMPLING

### GENERAL

Sample collection is an laboratory function. The Quality Control Officer interfaces with sample collectors to assure understanding of selection, collection and storage and transportation practices. For data to be meaningful, a sample must be representative of existing conditions and properly handled to avoid contamination or deterioration before it reaches the laboratory.

For routine activities, the location of each sampling site is selected and the number, type and size of containers is specified before a sample collector goes to the field. The sample source, amount to be collected, analyses and instructions are indicated on the sample container. Other pertinent information is written in waterproof, ballpoint, ink on a sample tag: sample number, date and time taken, source of sample, preservative, analyses to be performed and name of sample collector. All sampling follows techniques in the EPA Water Surveillance Branch Standard Operating Procedures and Quality Assurance Manual.

### Environmental Samples

Composite or grab sampling requirements are predetermined. Composite samples are preferred when "average concentrations" are the objective (e.g., wastewater treatment plant efficiency determinations); grab samples are preferred when the objective is documenting the extent, frequency and duration of variations in the sample. In some cases, both composite and grab sampling data are required.

All sample containers are sealed and tagged before they are shipped to the laboratory. Shipping procedures vary with the analyses to be performed. Refrigeration, as with ice or freeze packs, is used for samples for biochemical oxygen demand (BOD), phenol, nitrogen, cyanide, oil or chlorinated hydrocarbon analyses. Appropriate labels as "Do Not Freeze," or "Fragile," are used when applicable.

A field notebook (logbook) is completed at the site by the sample collector. Information equivalent to that on the tag is included, together with results of measurements taken in the field (e.g., temperature, dissolved oxygen [DO], pH, conductivity, flow) and pertinent observations at the time of sampling. Chain-of-custody forms are completed in the field at the time of sample collection and accompany the samples to the laboratory.

#### Industrial Hygiene Samples

NIOSH or other recognized procedures are used for sample collection, preservation and storage.

Sampling equipment and sampling media utilized are approved for industrial hygiene samples.

Sampling times and rates are as required by the methodology protocol. Pumps are calibrated in the laboratory immediately prior to use.

#### CONTINUOUS SAMPLING

- a. The accuracy of devices used for continuous sampling of liquids and gases involving the measurement of sample flow area rates and/or sample volume is determined on regularly scheduled basis.
- b. Adjustment of these devices is made as needed to bring the performance of the devices within specified limits.
- c. The frequency of sampling device calibration is specified and is based on the required accuracy, purpose, degree of usage, stability characteristics and other conditions affecting the measurement.
- d. Tests are conducted to verify that continuous sampling is representative of the material volumes sampled.

#### GRAB SAMPLING

- a. Tests are conducted to verify that grab samples are representative of the material sampled.
- b. Replicate samples are taken periodically to demonstrate the reproducibility of sampling.

#### MAINTENANCE OF SAMPLE INTEGRITY

Procedures for sampling, packaging, shipping and storage of samples provide for the maintenance of the integrity of the samples.

VII. QUALITY ASSURANCE/QUALITY CONTROL (QA/QC)  
PLAN FOR TREATABILITY STUDIES

PROGRAM DESIGN

Before any treatability and the associated analytical work can be performed, a program goal must be developed for that specific project. Once the goal has been defined, a program or Scope of Work is designed to meet it. Strict QA/QC measures will be used to monitor the program and ensure that all data generated are suitable for their intended use. The Treatability Study Manager and the Laboratory Director will be responsible to ensure that the QA/QC measures are properly employed.

The QA/QC plan for Kiber is essentially divided into two major groups; a general program plan and a project specific plan.

The general program plan sets up basic laboratory policies including QA/QC and standard operating procedures for specific tests. This serves as an operational charter for the laboratory.

The project specific plan differs from the program plan in that specific details of a particular treatability study are addressed in coordination with the client's requirements.

Project Organization

The samples received from the client initially will be checked for leakage and compared with the Chain of Custody document. The samples are then logged in Kiber's treatability study log book as required by the Treatability Ruling. The following is recorded for each sample:

- Client or contract
- Location of sampling
- Quantity of sample and type of material
- Number of designation of samples taken
- Date and time of collection
- Type of treatability study to be performed

## Treatability Study Operational Procedures

When performing treatability studies, Kiber is typically provided with samples from a given site. These samples usually have been collected by site personnel and are intended to be representative of the site materials. The accuracy of the treatability study, however, is only as good as the accuracy of the sample taken in the field; since Kiber has no control over the sample collection, the results of the study are assumed to be only estimations of anticipated results.

In an attempt to simulate site conditions for testing purposes, Kiber must take certain precautions when the samples are received at the laboratory. Initially, the entire sample is mixed in order to eliminate any "hot spots" that may exist and to ensure adequate blending of inconsistent materials. A sample of the untreated sample is then analyzed and compared to any data already available to ensure that the sample is representative of the site prior to proceeding with the study.

The QA/QC procedures of the specific treatability study are totally dependent on the type of study being performed, i.e. fixation, stabilization, water treatment, sludge dewatering, etc. Therefore, the Project Specification Plan is developed based on information provided by the client and can be submitted at the onset of the project if requested. As an example, Kiber will randomly select certain treatability mixtures for duplicate mixing and/or analysis to ensure consistency of operations.

### VIII. GENERAL WORK INSTRUCTIONS AND PROCEDURES

General work instructions and procedures used in all chemical analyses are written or referenced in an on-site work plan and made available to all analysts. The department supervisor is responsible for assuring that each analyst and technician is familiar with it and all pertinent operating procedures. Applicable instructions and procedures include:

- a. General rules
- b. Records
- c. Cleaning
  - (1) Glass and plastic laboratory ware
  - (2) Absorption cells
  - (3) Stopcock grease removal
- d. Weighing and balance care
- e. Standard samples
- f. Reagents storage and handling
- g. Operations
  - (1) General
  - (2) Gravimetric
  - (3) Volumetric
- h. Calibration
  - (1) Electrodes
  - (2) pH meter
  - (3) Other
- i. Quality control
- j. Laboratory safety

## IX. LABORATORY EQUIPMENT

### GENERAL

All equipment is maintained in proper working order with a written log for maintenance, repair and calibration of each appropriate piece of equipment. All major instrumentation is under service contract with the manufacturer and provided with required maintenance at regular intervals. Where applicable, reference materials certified by the National Bureau of Standards, including the thermometers, are used for calibration purposes. Kiber maintains the operating, service and calibration manuals provided by the manufacturer for all laboratory equipment. Maintenance files and service records are maintained for all instruments. Kiber meets or exceeds certification requirements regarding mandatory calibration and/or maintenance of equipment.

## X. CHEMICALS/REAGENTS

Chemicals no less pure than "analytical reagent grade" are used in the laboratory. A higher quality grade chemical or reagent is used when required by a particular procedure. "Pesticide grade" organic solvents are used for the organic extractions, and acids suitable for mercury determinations are used for metal analyses. Reagent blanks, methods blanks and working reagents are made with reverse osmosis/deionized water having a specific conductance of less than 2.0 micromhos/cm at 25 degrees Celsius.

Chemical standards are traceable to NBS Standards where traceability is possible. All standards used are certified standards or are ACS reagents quality materials.

## XI. METHODOLOGY

Analytical methods used by Kiber are published in Standard Methods for the Examination of Water and Wastewater, 15th Edition, 1980, 16th Edition, 1985, and 17th Edition, 1989; Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-20, 1979, (revised 1983); Method for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057, July 1982; Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, USEPA, 1982; Proposed Sampling and Analytical Methodologies for Evaluating Solid Waste Physical/Chemical Methods, 2nd Edition, USEPA, 1986; Federal Register, 40 CFR, Part 136, Volume 49, No. 209, October 26, 1984; Official Methods of Analysis of the Association of Analytical Chemists, 14th Edition, 1984; Annual Book of ASTM Standards, Water and Environmental Technology, Volume I and II, 1990; and NIOSH Manual of Analytical Methods, 3rd Edition, 1985.

All analyses are performed by use of standard procedures. As a general methodology guide, Standard Methods, published jointly by the American Public Health Association (APHA), American Water Works Association (AWWA), and Water Pollution Control Federation (WPCF), is used for environmental samples. The review requirement associated with this text (i.e., at intervals no greater than five years) assures current, on-going evaluating of adequacy and appropriateness of the methods. By definition, a "standard" method in this text is one that has undergone a structural program of collaborative testing; a "tentative" method is one that has been used with a high level of confidence, but has not been collaboratively tested.

EPA publishes the Manual of Chemical Methods which contains valid, rapid test methods having sufficient precision, accuracy and specificity to measure specific parameters in the presence of common interferences, using the skills and equipment that are usually available in water and wastewater laboratories. Additional EPA methods are published periodically in the Federal Register and are reviewed and adopted by Kiber.

Industrial hygiene procedures utilized are as published by the U.S. Department of Health, Education and Welfare, NIOSH Manual of Analytical Methods, or other recognized analytical procedure.

ASTM methods are used when applicable. All reported analyses reference the specific procedure that was followed. If data are to be used to support National Pollutant Discharge Elimination System permit requirements, compliance with stream or effluent quality standards or enforcement actions, EPA-approved methods are used. These methods are published from time to time in the Federal Register. A list of the methods routinely used by Kiber follows.

## XII. QUALITY CONTROL IN ANALYSIS

### GENERAL

All analysts practice quality control routinely. Routine quality control practices are established as part of every analytical measurement to assure reliability of final results. Every variable that can affect results is considered, evaluated and controlled; i.e., quality of reagent water, precision and accuracy of the analysis, daily performance of equipment and the analyst, and ongoing laboratory review.

Quality control requirements are met or exceeded for analyzing water as specified by pertinent certification requirements. All quality control data and performance records are maintained and available for inspection by the Client.

### INTRALABORATORY QUALITY CONTROL

Intralaboratory quality assurance is performed as described in the Handbook for Analytical Quality Control in Water and Wastewater Laboratories (EPA-600/4-79-019), Manual of Analytical Quality Control for Pesticides in Human Environmental Media (EPA-600/1-76-017) and the American Industrial Hygiene Association (AIHA) Quality Control Manual. Quality control charts for precision and accuracy have been developed and are used to ensure data reliability in accordance with the AIHA and the EPA manuals. Tabulated quality control data are reviewed by the Quality Control Officer and the analyst. Each analyst is provided with a quality control number which identifies him with each analysis he performs.

Duplicate samples are run for every tenth analysis of any given constituent. If any difference between duplicate analyses exceeds critical range value, then analyses are stopped until the problem is identified and resolved. After resolution, the problem and its solution are documented, and all analyses since the last in-control check are repeated or discarded. A known amount of standard sample of the constituent of interest is added on

a 1:1 concentration basis to every tenth analysis of any given parameter. In applying the accuracy control chart, either of the following two conditions would indicate an out-of-control situation:

- a. Any point beyond the control limits.
- b. Seven successive points on the same side of the average percent recovery line.

When an out-of-control situation occurs, analyses are stopped until the problem has been identified and resolved. The problem and its solution are documented, and all analyses since the last in-control point are repeated or discarded.

#### GC/MS QUALITY CONTROL

Gas Chromatograph/Mass Spectrometer analyses for priority pollutants are performed in accordance with EPA Methods 624, Volatile Organics and 625, Base/Nutrients, Acids and Pesticides published in the Federal Register, Vol. 44, No. 233, Monday, December 3, 1979. Details of the quality control procedures are discussed below.

a. Sample Preparation. The extraction procedure is critical to the quality of the work produced by Kiber. Technicians are thoroughly trained in the specific extraction and cleanup procedures to be used, as well as proper utilization and maintenance of the Kuderna-Danish glassware.

b. Mass Calibration. At the beginning of each eight hour shift the mass calibration of the system is checked by injecting a solution of BFB, if volatiles are being analyzed, or DFTPP if extractable analyses are to be performed. Key ions and ion abundance criteria are listed in table 1. A listing of the calibration ions is retained as documentation that the calibration was performed and was within specifications.

c. Analyte Calibration. The GC/MS system is initially calibrated at a minimum of three concentrations of all the priority pollutants to determine response factors as described in Section 6 of Method 624 or 625. Once the initial calibration has been performed, the calibration is verified at least once eight hour shift by analyses of

representative compounds from each fraction. If the mean percent change for these calibration check compounds is greater than twenty percent, the system is recalibrated using a minimum of three concentrations for each compound. Response factors for the calibration check compounds are recorded daily and monitored for signs of change in the performance of the system.

$$\text{Percent Change} = \frac{\text{RF Check} - \text{RF Initial}}{\text{RF Initial}} \times 100\%$$

d. Surrogate Standards. Before each sample is extracted a minimum of two surrogate standards are added to produce a final concentration of 50 g/l. The sample is then processed through the entire procedure including GC/MS analyses. If the recovery of the surrogate standard shows a deviation of greater than two standard deviations, the data are suspect and supervisory personnel must investigate the cause of the error.

e. Blank Analyses. A reagent blank analysis is performed either once per sample set or once per 20 samples and whenever a new lot of reagents is utilized in the analyses. If priority pollutants are detected in the blank, no analyses are performed until the source of the contamination can be determined.

f. Replicate Analyses. A minimum of one in twenty samples is a duplicate analyses. The results of this analysis give a value for the precision of the method under normal operating conditions.

g. Spike Samples. A minimum of one in twenty samples is spiked with a mixture of priority pollutants and analyzed in the same manner as other samples. The percent recovery for each compound is calculated to provide an indication of the accuracy of the method for priority pollutant analyses under typical operating conditions.

h. Outliers. Outliers are defined as any result greater than three standard deviations from the mean ( $R+3S$ ) for both duplicate and spiked results. Corrective action outliers involves one or more of the following: repreparation and reanalysis, recalculation of the sample based on alternate ions, preparation of new standards, and recalibration of the instrument. Documentation of the corrective action is included in the laboratory notebook.

Table 1 DFTPP/BFB Key Ions and Abundance Criteria

<u>Mass</u>	<u>Specifications - DFTPP</u>
51	30 to 60 percent of mass 198
68	Less than 2 percent of mass 69
70	Less than 2 percent of mass 69
127	40 to 60 percent of mass 198
197	Less than 1 percent of mass 198
198	Base peak, 100 percent relative abundance
199	5 to 9 percent of mass 198
275	10 to 30 percent of mass 198
365	Greater than 1 percent of mass 198
441	Present but less than mass 443
442	Greater than 40 percent of mass 198
443	17 to 23 percent of mass 442

<u>Mass</u>	<u>Specifications - BFB</u>
50	15 to 40 percent of mass 95
75	30 to 60 percent of mass 95
95	Base peak, 100 percent relative abundance
96	5 to 9 percent relative abundance
173	Less than 2 percent of mass 174
174	Greater than 50 percent of mass 95
175	5 to 9 percent of mass 174
176	Greater than 95 and less than 101 percent of mass 174
177	5 to 9 percent of mass 176

i. Archival Storage. All GC/MS sample data are stored on 9 track magnetic tape in EPA readable format. The archival storage of data allows samples to be re-analyzed at any time, providing proof of previous identifications, and the ability to search for other non-priority pollutant compounds.

### XIII. DATA REDUCTION AND REPORTING

All laboratory data are either hard copied by computer printouts or entered into bound notebooks by the analyst for permanent storage. Data reduction is either performed manually by the analyst in accordance with method calculations or is performed by data stations interfaced with the analytical instruments.

All data are reviewed by four different levels of laboratory personnel. The review process includes the analyst, supervisor (section chief), quality control officer and chief chemist. Out-of-control conditions are identified by the analyst, supervisor or quality control officer; investigated, corrected, and documented. Documentation of the corrective action is signed by the quality control officer. The 99 percent confidence interval is used to determine out-of-control conditions.

Quality control charts are maintained by each laboratory section and area accessible to all analysts.

REQUIRED CONTAINERS, PRESERVATION  
AND HOLDING TIMES

Parameter	Container	Preservation	Maximum Holding Time
Coliform Bacteria	P,G	Cool, 4C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine is present	6 hours
Fecal Streptococci	P,G	do	do
Acidity	P,G	Cool, 4C	14 days
Ammonia	P,G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
BOD	P,G	Cool, 4C	48 hours
Bromide	P,G	do	28 days
COD	P,G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	do
Chloride	P,G	Cool, 4C	do
Chlorine, total residual	P,G	None required	Analyze immediately
Color	P,G	do	48 hours
Cyanide	P,G	Cool, 4C, NaOH to pH>12 add ascorbic acid if residual chlorine is present	14 days
Fluoride	P	Cool, 4C	28 days
Hardness	P,G	HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months
pH	P,G	None required	Analyze immediately
Kjeldahl Nitrogen	P,G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Chromium VI	P,G	Cool, 4C	24 hours
Mercury	P,G	HNO <sub>3</sub> to pH<2	28 days
All other metals	P,G	do	6 months
Nitrate	P,G	Cool, 4C	48 hours
Nitrate-nitrite	P,G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days

Parameter	Container	Preservation	Maximum Holding Time
Nitrite	P,G	Cool, 4C	48 hours
Oil & Grease	G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
TOC	P,G	do	do
Orthophosphate	P,G	Filter immediately, Cool, 4C	48 hours
Dissolved Oxygen	G	None required	Analyze immediately
Phenols	G	Cool, 4C, H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
Total Phosphorus	P,G	do	do
Total Residue	P,G	Cool, 4C	7 days
TDS	P,G	do	48 hours
TSS	P,G	do	7 days
Silica	P,G	do	28 days
Spec. Conductance	P,G	do	do
Sulfate	P,G	do	do
Sulfide	P,G	Cool, 4C, add zinc acetate plus NaOH to pH>9	7 days
Sulfite	P,G	None required	Analyze immediately
Surfactants	P,G	Cool, 4C	48 hours
Turbidity	P,G	do	do
VOCs	G, Teflon-lined septum	Cool, 4C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine is present, HCl to pH <2	14 days
Extractable Organics	G, Teflon-lined caps	Cool, 4C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> if residual chlorine is present	7 days until extraction, 40 days after extraction
Radiological Tests	P,G	HNO <sub>3</sub> to pH<2	6 months

P = plastic  
G = glass  
do = Ditto

Source: 40 CFR Part 136, 1991

TABLE 1A - LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

Parameter and Units	Method	Standard Methods, 15th Ed.	SW 846
Bacteria:			
1. Coliform (fecal) number per 100 mL	Membrane filter (MF)	980C	
2. Coliform (total) number per 100 mL	MF single step or two step	908A, 909A	9132
3. Fecal streptocci, number per 100 mL		910A	

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
1. Acidity, as CaCO <sub>3</sub> , mg/L: Electrometric end point or phenolphthalein end point	305.1	402(4.d)	D1067-70(E)	
2. Alkalinity, as CaCO <sub>3</sub> , mg/L: Electrometric or colorimetric: Titration to pH 4.5, manual or automated	310.1 310.2	403	D1067(B)	
3. Aluminum--Total, mg/L: Digestion followed by: AA direct aspiration AA furnace Inductively coupled plasma	202.1 202.2 200.7	303C 304		6010
4. Ammonia (as N), mg/L: Manual distillation (at pH 9.5): Followed by Titration Electrode Automated phenate, or Automated electrode	350.2 350.2 350.3 350.1	417A 417D 417F	D1426-79(D) D1426-79(C)	
5. Antimony--Total, mg/L: Digestion followed by: AA direct aspiration AA furnace, or Inductively coupled plasma	204.1 204.2 200.7	303A 304		7040 7041 6010
6. Arsenic--Total, mg/L: Digestion followed by Hydride AA furnace Inductively coupled plasma	206.5 206.3 206.2 200.7	303E 304	D2972-78(B)	7061 7060
7. Barium--Total, mg/L: Digestion followed by: AA direct aspiration AA furnace or Inductively coupled plasma	208.1 208.2 200.7	303C 304		7080 7081 6010

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
8. Beryllium—Total, mg/L: Digestion followed by:				
AA direct aspiration	210.1	303C	D3645-78	7090
AA furnace	210.2	304		7091
Inductively coupled plasma	200.7			6010
9. Biochemical oxygen demand (BOD5), mg/L:				
Winkler (Azide modification)	405.1	507		
Or electrode method				
10. Boron—Total, mg/L:				
Colorimatic (curcumin or	212.3	404A		
Inductively coupled plasma	200.7			6010
11. Bromide, mg/L: Titrimetric	320.1		D1246-77(C)	
12. Cadmium—Total, mg/L: Digestion followed by:				
AA direct aspiration	213.2	303A or 303B	D3557-78(A or B)	7130
AA furnace	213.2	304		7131
Inductively coupled plasma	200.7			6010
13. Calcium—Total, mg/L: Digestion followed by:				
Atomic absorption	215.1	303A	D511-77(C)	6010
Inductively coupled plasma	200.7			
Or EDTA titration	215.2	311C	D511-77(B)	
14. Carbonaceous biochemical oxygen demand (CBOD5), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor		507(5.e.6)		
15. Chemical oxygen demand (COD), mg/L: Spectrophotometric		Oceanography International Corporation, ampule method		
16. Chloride, mg/L:				
Titrimetric mercuric nitrate	325.3	407B	D512-67(A)	9252
Colorimetric (ferricyanide) automated	325.1 or 325.2	407D		9250
17. Chlorine—Total residual, mg/L: Titrimetric-ampereometric	330.1	408C	D1253-76(A)	

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
18. Chromium VI dissolved, mg/L: 0.45 micron filtration with:				
Extraction and atomic absorption, or Colorimetric (Diphenylcarbazide)	218.4	303B		7197 7196
19. Chromium--Total, mg/L:				
Digestion (optional extraction) followed by	218.3			
AA direct aspiration	218.1	303A or 303B	D1687-77(D)	7190
AA furnace	218.2	304		7191
Inductively coupled plasma	200.7			6010
20. Cobalt--Total, mg/L: Digestion followed by:				
AA direct aspiration	219.1	303A or 303B	D3558-77(A or B)	
AA furnace, or	219.2	304		
Inductively coupled plasma	200.7			6010
21. Color, platinum cobalt units or dominant wave-length hue, luminance, purity:				
Platinum cobalt; or	110.2	204A		
Spectrophotometric	110.3	204B		
22. Copper--Total, mg/L: Digestion followed by:				
AA direct aspiration	220.1	303A or 303B	D1688-77(D or E)	7210
AA furnace	220.2	304		7211
Inductively coupled plasma	200.7			6010
23. Cyanide--Total, mg/L:				
Manual distillation with MgCl <sub>2</sub>	335.2	412D		
Followed by titrimetric	335.2	412B		
Manual or	335.2	412C	D2036-75(A)	
Automated spectrophotometric	335.3	412D	D2036-75(A)	
24. Cyanide amenable to chlorination, mg/L: Manual distillation with MgCl <sub>2</sub> : Followed by titrimetric, manual or automated spectrophotometric	335.1	412F	D2036-75(B)	

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
25. Fluoride--Total, mg/L: Manual distillation Followed by manual or Automated electrode	340.2	413A 413B	D1179-72(B)	
26. Gold--Total, mg/L: Digestion followed by: AA direct aspiration Or AA furnace	231.1 231.2	303A 304		
27. Hardness--Total as CaCO <sub>3</sub> , mg/L: EDTA titration Inductively coupled plasma Or atomic absorption (sum of Ca and Mg as their respective carbonates)	130.2 200.7 251.1 + 242.1	314B 303A	D1126-67(B)	
28. Hydrogen ion (pH), pH units: Electrometric	150.1	423	D1293-78(A) or D-1293-78(B)	9040
29. Ignitability closed cup, degrees C:			D93-77	1010
30. Indium--Total, mg/L: Digestion AA direct aspiration Or AA furnace	235.1 235.2	303A 304		
31. Iron--Total, mg/L: Digestion followed by AA direct aspiration AA furnace Inductively coupled plasma Or colorimetric (Phenanthroline)	236.1 236.2 200.7	303A or 303B 303B 304 315B	D1068-77 (C or D) D1068-77(A)	7380 7381 6010
32. Kjeldahl nitrogen--Total (as N), mg/L: Digestion and distillation Followed by titration Electrode	351.3 351.3 351.3	420A or B 417D 417E	D3590-77	

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
33. Lead--Total, mg/L: Digestion followed by:				
AA direct aspiration	239.1	303A or 303B	D3559-78(A or B)	7420
AA furnace	239.2	304		7421
Inductively coupled plasma	200.7			6010
34. Magnesium--Total, mg/L: Digestion followed by:				
Atomic absorption	242.1	303A	D511-77(B)	
Inductively coupled plasma				6010
35. Manganese--Total, mg/L: Digestion followed by:				
AA direct aspiration	243.1	303A or 303B	D858-77(B or C)	7460
AA furnace	243.2	304		7461
Inductively coupled plasma	200.7			6010
36. Mercury--Total, mg/L: Cold vapor, manual	245.1	303F	D3223-79	7470
37. Molybdenum--Total, mg/L: Digestion followed by:				
AA direct aspiration	246.1	303C		
AA furnace, or	246.2	304		
Inductively coupled plasma	200.7			6010
38. Nickel--Total, mg/L: Digestion followed by:				
AA direct aspiration	249.1	303A or 303B	D1886-77(C or D)	7520
AA furnace	249.2	304		7521
Inductively coupled plasma	200.7			6010
39. Nitrate (as N), mg/L: Nitrate-nitrite N minus Nitrite N	See para- meters 39 & 40	See para- meters 39 & 40	See para- meters 39 & 40	
40. Nitrate-nitrite (as N), mg/L: Cadmium reduction, automated	353.2	418F	D3867-79(A)	
41. Nitrite (as N), mg/L: Spectrophotometric, Automated (Diazotization)	354.1	419	D1254-67	
42. Oil and grease--Total recoverable, mg/L: Gravimetric (extraction)	413.1	503A		9070

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
43. Organic carbon--Total (TOC), mg/L: Combustion or oxidation	415.1	505	D2579-78(A) or D2579-78(B)	9060
44. Organic halides--Total (TOX),ug/L	450.1			9020
45. Organic nitrogen (as N), mg/L: Total kjeldahl N minus ammonia N	See para- meters 32 & 4	420A	D3590-77 minus D1426-79(A)	
46. Orthophosphate (as P), mg/L: Ascorbic acid method, automated	365.1	424G		
47. Osmium--Total, mg/L: Digestion followed by: AA direct aspiration or AA furnace	252.1 252.2	303C 304		7550 7551
48. Oxygen, dissolved, mg/L: Winkler (Azide modification) Or electrode	360.2 360.1	421B 421F	D1589-60(A)	
49. Palladium--Total, mg/L: Digestion followed by: AA direct absorption Or AA furnace	253.1 253.2			
50. Phenols, mg/L: Manual distillation Followed by manual colorimetric (4AAP)	420.1 420.1		D1783-70(A or B)	9065 9065
51. Phosphorous--Total, mg/L: Persulfate digestion Followed by automated ascorbic acid	365.2 365.1	424C(III) 424G		
52. Platinum--Total, mg/L: Digestion followed by: AA direct aspiration Or AA furnace	255.1 255.2	303A 304		
53. Potassium--Total, mg/L: Digestion followed by: Atomic absorption Inductively coupled plasma	258.1 200.7	303A		6010
54. Residue--Total, mg/L: Gravimetric, 103-105C	160.3	209A		
55. Residue--filterable, mg/L: Gravimetric, 180C	160.1	209B		

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
56. Residue--nonfilterable (TSS), mg/L: Gravimetric, 103-105C post washing of residue	160.2	209D		
57. Residue--settleable, mg/L: Volumetric (Imhoff cone) or gravimetric	160.5	209F		
58. Residue--volatile, mg/L: Gravimetric 550 degrees celcius	160.4	209E		
59. Rhodium--Total, mg/L: Digestion followed by: AA direct aspiration Or AA furnace	265.1 267.2	303A 304		
60. Ruthenium--Total, mg/L: Digestion followed by: AA direct aspiration Or AA furnace	267.1 267.2	303A 304		
61. Selenium--Total, mg/L: Digestion followed by: AA furnace Inductively coupled plasma Or hydride	270.2 200.7 270.3	304 303E	D3859-79	7740 7741
62. Silica--Dissolved, mg/L: 0.45 micron filtration: Followed by inductively coupled plasma	200.7			6010
63. Silver--Total, mg/L: Digestion followed by: AA direct aspiration AA direct furnace or Inductively coupled plasma	272.1 272.1 200.7	303A or 303B 304		7760 7761 6010
64. Sodium--Total, mg/L: Digestion followed by: Atomic absorption Inductively coupled plasma	273.1 200.7	303A		7770 6010
65. Specific conductance, mhos/cm: Wheat- stone bridge	120.1	205	D1125-77(A)	

TABLE 1B - LIST OF APPROVED INORGANIC TEST PROCEDURES

PARAMETERS, UNITS AND METHOD	EPA 1979, 1983 Revision	Reference (Method No.)		
		Standard Methods, 15th Ed.	ASTM	SW 846
66. Sulfate (as SO <sub>4</sub> ), mg/L: Automated methylthymol blue Gravimetric or Turbidimetric	375.2			9036
	375.3	426A or 426B	D516-68(A)	9037
	375.4	426C	D516-68(B)	9038
67. Sulfide (as S), mg/L: Titrimetric (iodine) or Colorimetric (methylene blue)	376.1	427D		9030
	376.2	427C		
68. Sulfite (as SO <sub>3</sub> ), mg/L: Titrimetric (iodine iodate)	377.1	428F	D1339-78(C)	
69. Surfactants, mg/L: Colorimetric (methylene blue)	425.1	512A	D2330-68(A)	
70. Temperature, in degrees celcius: Thermometric	170.1	212		
71. Thallium--Total, mg/L: Digestion followed by:				
	AA direct aspiration	279.1	303A	7840
	AA furnace, or	279.2	304	7841
	Inductively coupled plasma	200.7		6010
72. Tin--Total, mg/L: Digestion followed by:				
	AA direct aspiration	282.1	303A	
AA furnace	282.2	304		
73. Titanium--Total, mg/L: Digestion followed by:				
	AA direct aspiration or	283.1	303C	
AA furnace	283.2	304		
74. Turbidity, NTU: Nephelometric	180.1	214A	D1889-71	
75. Vanadium--Total, mg/L: Digestion followed by:				
	AA direct aspiration	286.1	303C	7910
	AA furnace	286.2	304	7911
	Inductively coupled plasma	200.7		6010
76. Zinc--Total, mg/L: Digestion followed by:				
	AA direct aspiration	289.1	303A or 303B	D1691-77(D)
	AA furnace	289.2	304	D1691-77(C)
	Inductively coupled plasma	200.7		6010

TABLE IC - LIST OF APPROVED TEST PROCEDURES  
FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter	EPA METHOD NUMBER		
	GC	GC/MS	HPLC
1. Acenaphthene	610	8250, 625, 1625	8310, 610
2. Acenaphthylene	610	8250, 625, 1625	8310, 610
3. Acrolein	8030, 603	8250, 624, 1624	
4. Acrylonitrile	8030, 603	8250, 624, 1624	
5. Anthracene	610	8250, 625, 1625	8310, 610
6. Benzene	602	8240, 624, 1624	
7. Benzidine		8250, 625, 1625	605
8. Benzo(a)anthracene	610	8250, 625, 1625	8310, 610
9. Benzo(a)pyrene	610	8250, 625, 1625	8310, 610
10. Benzo(b)fluoranthene	610	8250, 625, 1625	8310, 610
11. Benzo(ghi)perylene	610	8250, 625, 1625	8310, 610
12. Benzo(k)fluoranthene	610	8250, 625, 1625	8310, 610
13. Benzyl Chloride			
14. Benzyl Butyl Phthalate	606	8250, 625, 1625	
15. Bis(2-chloroethoxy) methane	611	8250, 625, 1625	
16. Bis(2-chloroethyl) ether	611	8250, 625, 1625	
17. Bis(2-ethylhexyl) Phthalate	606	8250, 625, 1625	
18. Bromodichloromethane	601	8240, 624, 1624	
19. Bromoform	601	8240, 624, 1624	
20. Bromomethane	601	8240, 624, 1624	
21. 4-Bromophenylphenyl ether	611	8250, 625, 1625	
22. Carbon tetrachloride	601	8240, 624, 1624	
23. 4-Chloro-2-methylphenol	604	8250, 625, 1625	
24. Chlorobenzene	601, 602	8240, 624, 1624	
25. Chloroethane	601	8240, 624, 1624	
26. 2-Chloroethylvinyl ether	601	8240, 624, 1624	
27. Chloroform	601	8240, 624, 1624	
28. Chloromethane	601	8240, 624, 1624	
29. 2-Chloronaphthalene	612	8250, 625, 1625	
30. 2-Chlorophenol	604	8250, 625, 1625	
31. 4-Chlorophenylphenyl ether	611	8250, 625, 1625	
32. Chrysene	610	8250, 625, 1625	8310, 610
33. Dibenzo(a,h)anthracene	610	8250, 625, 1625	8310, 610
34. Dibromochloromethane	601	8240, 624, 1624	
35. 1,2-Dichlorobenzene	601, 602, 612	8240, 624, 625, 1625	
36. 1,3-Dichlorobenzene	601, 602, 612	8240, 624, 625, 1625	
37. 1,4-Dichlorobenzene	601, 602, 612	8240, 625, 1624, 1625	
38. 3,3-Dichlorobenzidine		8250, 625, 1625	605
39. Dichlorodifluoromethane	601		
40. 1,1-Dichloroethane	601	8240, 624, 1624	
41. 1,2-Dichloroethane	601	8240, 624, 1624	
42. 1,1-Dichloroethane	601	8240, 624, 1624	
43. trans-1,2-Dichloroethene	601	8240, 624, 1624	
44. 2,4-Dichlorophenol	604	8250, 625, 1625	
45. 1,2-Dichloropropane	601	8240, 624, 1624	
46. cis-1,3-Dichloropropene	601	8240, 624, 1624	

TABLE IC - LIST OF APPROVED TEST PROCEDURES  
FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter	EPA METHOD NUMBER		
	GC	GC/MS	HPLC
47. trans-1,3-Dichloropropene	601	8240, 624, 1624	
48. Diethyl phthalate	606	8250, 625, 1625	
49. 2,4-Dimethylphenol	604	8250, 625, 1625	
50. Dimethyl phthalate	606	8250, 625, 1625	
51. Di-n-butyl phthalate	606	8250, 625, 1625	
52. Di-n-octyl phthalate	606	8250, 625, 1625	
53. 2,4-Dinitrophenol	604	8250, 625, 1625	
54. 2,4 Dinitrotoluene	609	8250, 625, 1625	
55. 2,6-Dinitrotoluene	609	8250, 625, 1625	
56. Epichlorohydrin			
57. Ethylbenzene	602	8240, 624, 1624	
58. Fluoranthene	610	8250, 625, 1625	8310, 610
59. Fluorene	610	8250, 625, 1625	8310, 610
60. Hexachlorobenzene	612	8250, 625, 1625	
61. Hexachlorobutadiene	612	8250, 625, 1625	
62. Hexachlorocyclopentadiene	612	8250, 625, 1625	
63. Hexachloroethane	612	8250, 625, 1625	
64. Ideno(1,2,3-cd)pyrene	610	8250, 625, 1625	8310, 610
65. Isophorone	609	8245, 625, 1625	
66. Methylene Chloride	601	8240, 624, 1624	
67. 2-Methyl-4,6-Dinitrophenol	604	8250, 625, 1625	
68. Naphthalene	610	8250, 625, 1625	8310,610
69. Nitrobenzene	609	8250, 625, 1625	
70. 2-Nitrophenol	604	8250, 625, 1625	
71. 4-Nitrophenol	604	8250, 625, 1625	
72. N-Nitrosodimethylamine	607	8250, 625, 1625	
73. N-Nitrosodi-n-propylamine	607	8250, 625, 1625	
74. N-Nitrosodiphenylamine	607	8250, 625, 1625	
75. 2,2-oxybis(1-chloropropane)	611	8250, 625, 1625	
76. PCB-1016	8080, 608	8250	625
77. PCB-1221	8080, 608	8250	625
78. PCB-1232	8080, 608	8250	625
79. PCB-1242	8080, 608	8250	625
80. PCB-1248	8080, 608	8250	625
81. PCB-1254	8080, 608	8250	625
82. PCB-1260	8080, 608	8250	625
83. Pentachlorophenol	604	8250, 625, 1625	
84. Phenanthrene	610	8250, 625, 1625	8310, 610
85. Phenol	604	8250, 625, 1625	
86. Pyrene	610	8250, 625, 1625	8310, 610
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin (screen)		8250, 625	
88. 1,1,2,2-Tetrachloroethane	601	8240, 624, 1624	
89. Tetrachloroethene	601	8250, 625, 1625	
90. Toluene	602	8240, 624, 1624	
91. 1,2,4-Trichlorobenzene	612	8240, 625, 1625	
92. 1,1,1-Trichlorobenzene	601	8240, 624, 1624	
93. 1,1,2-Trichloroethane	601	8240, 624, 1624	

Parameter	EPA Method Number		
	GC	GC/MS	HPLC
94. Trichloroethene	601	8240, 624, 1624	
95. Trichlorofluoromethane	601	8240, 624	
96. 2,4,6-Trichlorophenol	604	8250,625, 1625	
97. Vinyl Chloride	601	8240, 624, 1624	

TABLE 1D - LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES

Parameter	Method	EPA	15th Ed.	ASTM
1. Aldrin	GC	8080,608	509A	D3086
	GC/MS	8250,625		
2. Ametryn	GC			
3. Aminocarb	TLC			
4. Atraton	GC			
5. Atrazine	GC			
6. Azinphos methyl	GC			
7. Barban	TLC			
8. -BHC	GC	8080,608	509A	D3086
	GC/MS	8250,625		
9. Beta -BHC	GC	8080,608		D3086
	GC/MS	8250,625		
10. -BHC	GC	8080,608		D3086
	GC/MS	8250,625		
11. -BHC (Lindane)	GC	8080,608	509A	D3086
	GC/MS	8250,625		
12. Captan	GC		509A	
13. Carbaryl	TLC			
14. Carbophenothion	GC			
15. Chlordane	GC	8080,608	509A	D3086
	GC/MS	8250,625		
16. Chloroprotham	TLC			
17. 2,4-D	GC	8150	509B	
18. 4,4-DDD	GC	8080,608	509A	D3086
	GC/MS	8250,625		
19. 4,4-DDE	GC	8080,608	509A	D3086
	GC/MS	8250,625		
20. 4,4-DDT	GC	8080,608	509A	D3086
	GC/MS	8250,625		
21. Demeton-O	GC			
22. Demeton-S	GC			
23. Diazinon	GC	8140		
24. Dicamba	GC	8150		
25. Dichlofenthion	GC			
26. Dichloran	GC		509A	
27. Dicofol	GC			D3086
28. Dieldrin	GC	8080,608	509A	
	GC/MS	8250,625		
29. Dioxathion	GC			

Parameter	Method	EPA	15th Ed.	ASTM
30. Disulfoton	GC	8140		
31. Diuron	TLC			
32. Endosulfan I	GC	8080, 608	509A	D3086
	GC/MS	8250, 625		
33. Endosulfan II	GC	8080, 608	509A	D3086
	GC/MS	8250, 625		
34. Endosulfan sulfate	GC	8080, 608		
	GC/MS	8250, 625		
35. Endrin	GC	8080, 608	509A	D3086
	GC/MS	8250, 625		
36. Endrin aldehyde	GC	8080, 608		
	GC/MS	8250, 625		
37. Ethion	GC			
38. Fenuron	TLC			
39. Fenuron-TCA	TLC			
40. Heptachlor	GC	8080, 608	509A	D3086
	GC/MS	8250, 625		
41. Heptachlor epoxide	GC	8080, 608	509A	D3086
42. Isodrin	GC/MS	8250, 625		
	GC			
43. Linuron	TLC			
44. Malathion	GC		509A	
45. Methiocarb	TLC			
46. Methoxychlor	GC		509A	D3086
47. Mexacarbate	TLC			
48. Mirex	GC		509A	
49. Monuron	TLC			
50. Monuron-TCA	TLC			
51. Neburon	TLC			
52. Parathion methyl	GC	8140	509A	
53. Parathion ethyl	GC		509A	
54. PCNB	GC		509A	
55. Perthane	GC			D3086
56. Prometon	GC			
57. Prometryn	GC			
58. Propazine	GC			
59. Propham	TLC			
60. Propoxur	TLC			
61. Sebumeton	TLC			
62. Siduron	TLC			
63. Simazine	GC			
64. Strobane	GC		509A	
65. Swep	TLC			
66. 2,4,5-T	GC	8150	509B	
67. 2,4,5-TP(Silvex)	GC	8150	509B	
68. Terbutylazine	GC			
69. Toxaphene	GC	8080,608	509A	D3086
	GC/MS	8250, 625		
70. Trifluralin	GC		509A	

**APPENDIX B**  
**KIBER ANALYTICAL SERVICES, INC. - GC/MS PROCEDURES**

## **GAS CHROMATOGRAPHY/MASS SPECTROMETRY**

### **1.0 OVERVIEW**

The standard operating procedures for gas chromatography/mass spectrometry (GC/MS) are separated into three sections: GC/MS overview, volatiles analysis, and semivolatiles analysis. The GC/MS overview includes personnel responsibilities and software programs. Volatiles and semivolatiles analysis each include procedures for preparation, analysis, quality assurance and data reporting.

### **2.0 RESPONSIBILITIES AND CHAIN OF COMMAND**

#### **2.1 Analyst**

The analyst is responsible for the sample preparation, calibration, data acquisition/processing, and compilation of quality assurance data written under this operating procedure. If assistance is needed to carry out these functions, the analyst should consult with the GC/MS Supervisor and only with the Laboratory Director on matters requiring interlaboratory coordination.

#### **2.2 GC/MS Supervisor**

The GC/MS Supervisor is responsible for the overall operation of the GC/MS laboratory including training and assistance of analysts, initial data review, data reporting, sample throughput, quality assurance review, system troubleshooting and supplies. He may designate duties to the analysts if they are capable. If the GC/MS Supervisor needs assistance in the execution of his duties, he should consult with the Laboratory Director.

#### **2.3 Laboratory Director**

The Laboratory Director is responsible for the overall operation of the laboratory. The duties include initiation of the laboratory and procedures, technical and report review, spectral interpretation, coordination between organic sections, assessment of capital needs, safety and sample flow.

### 3.0 GC/MS SYSTEM SOFTWARE

Software inherent in the Hewlett-Packard RTE-A data system is used to operate and monitor the instruments, perform analyses, process analytical data and archive files for GC/MS operations. The software program descriptions, formats, entries and options are detailed below. Many utilize "soft key" operations through Aquarius software which, in most instances, can be more efficiently utilized by direct string command input. Soft keystrokes are denoted by fn, where n is the number designation of the soft key.

A basic familiarity with the operating system is assumed. The analyst can always consult the system manuals or the supervisor for familiarization.

#### 3.1 Tuning Programs

Tuning programs are utilized to manually or automatically tune with perfluorotributylamine (PFTBA) to criteria, which assist in satisfaction of Environmental Protection Agency (EPA) tuning requirements. The different methods of tuning, the general tuning processes and conventions for naming tuning files are discussed below.

Tuning is performed using the program MS Calibration (MSCAL). The program can be invoked by typing MSCAL or through the Tuning & Diag: System Tuning soft-key pathway. The MSCAL program provides two basic tuning options: Autotune and manual tune (MTUNE).

##### 3.1.1 Autotune

Autotune is an automated tuning program which automatically adjusts the mass spectrometer parameters to achieve certain predefined performance criteria. Autotune calculates the normalization factors required to make the spectra produced by the instrument consistent with the EPA requirements for DFTPP (decafluorotriphenylphosphine).

Autotune is primarily used in Kiber Analytical operations as a preliminary tuning process used before manual tune. It is used when the tune spectra produced is quite removed from the required criteria. This most often occurs after source cleaning and instrument modifications. Autotune is used sparingly with the volatile instruments since the high mass range is not monitored in the BFB tuning criteria.

### 3.1.1.1 Autotune Operation

Before tuning, **OPEN** the PFTBA calibration valve.

Autotune is a softkey and form-driven process. After entering MSCAL, press the Auto Tune soft-key, then press the soft-key of the instrument to be tuned. If one instrument is being used in a manner such that Autotune would be inappropriate to run at this time, the softkey display shows "IN USE" and the user is not able to access it for tuning. The Autotune run parameters are presented in a form.

### 3.1.1.2 Autotune form

The data entered on this form are as follows:

**Tune from Std/Previous values:** Enter P unless this is the first tuning process performed on this instrument or **major** alterations have been performed. In these cases enter S.

**Force tune:** Enter Y.

**Auto copy of Final report:** enter Y, **list to:** enter T.

**Trace report:** enter N.

**Spectrum plot:** enter N.

**Norm. spectrum:** enter N.

When the form is complete, press the Enter Data softkey. The Autotune process proceeds to completion, displays an error message or in some cases "hangs-up". When finished with tuning procedures, **TURN OFF THE PFTBA VALVE!**

### 3.1.1.3 Autotune results

Successful Autotunes produce a printed report showing header information, the tune settings, the masses and abundances, and messages. This information assists in diagnosing the status of the mass spectrometer. By comparing these values as they change over time, the analyst and/or supervisor can tell when the instrument performance starts to deteriorate. **All final reports generated by Autotune are retained.** Note parameter values, which can be used as a starting point in the manual tuning process. Note particularly the EMvlts (Electron Multiplier voltage) final setting as this (or a function of this) can be used as the EM voltage setting in the actual tuning files.

Unsuccessful Autotunes indicate system problems which should be resolved by troubleshooting. Common tuning problems result from a lack of tuning compound (empty supply or valve closed), insensitivity (too low of a starting EM voltage setting or source problems), or the instrument electronics being shut off.

### 3.1.2 Manual Tuning

Manual Tune (MTUNE) is used as the primary tuning procedure in Kiber Analytical operations. Manual tuning is entered by typing MTUNE after entering MSCAL. Softkeys are utilized throughout the remainder of the process. The manual tune softkeys are divided among three levels: TOP, whose function is essentially that of bookkeeping; EDIT, whereby parameters can be edited through forms mode interaction; and EXECUTE, enabling mass spectrometer operation and adjustments using softkeys. Manual tuning procedures and tuning aides are detailed after the descriptions of the softkeys.

#### 3.1.2.1 TOP level keys

The function of the top level softkeys are as follows:

**f1 (Read Autotune):** The purpose of this command is to bring the current Autotune file data from disc into active use. This file contains all the Autotune parameters generated during the Autotune procedure described previously.

**f2 (New Tune File):** Allows the user to create space and names for a custom tuning file generated during the manual tuning process or to read the parameters from a previously created file.

**f3 (Execute):** Pushing this key generates the softkeys of the EXECUTE level.

**f4 (Edit):** Pushing this key takes the operator to the EDIT level keys.

**f5 (not used)**

**f6 (Store Tune):** This command stores the current mass spectrometer parameters from MTUNE to a disc file. The tune parameters can be stored into an existing manual tune file, an Autotune file or a new file created by this command.

**f7 (System Commands)**: This softkey causes the standard System Command softkeys to be displayed. These keys allow a convenient way of making alpha and graphics copies, or screen displays, or cloning FMGR to determine a system status and other commands without having to exit the MTUNE program.

**f8 (Exit)**: This softkey allows the user to exit the MTUNE program and return to FMGR or to MSCAL, depending on the level from which MTUNE was scheduled.

#### 3.1.2.2 EDIT level keys

The edit level of softkeys allows the user to examine existing parameters and to make modifications in establishing a tuning file. The edit level keys are:

**f1 (Tune Params)**: Allows the operator to examine existing instrument parameters and to make desired changes.

**f2 (Scan Params)**: Shows the scanning parameters for a typical tuning file.

**f3 (Ramp Params)**: This form shows the limits and step sizes for the various mass spectrometer parameters. These values are used for tuning with the repeat profile and parameter ramp EXECUTE options.

**f4, f5, f6, f7 (not used)**

**f8 (Return to MTUNE)**: Moves the operator to the previous level of softkeys.

### 3.1.2.3 EXECUTE level key

There are two alternating sets of softkeys within EXECUTE. The first set is reached when moving from the Execute key at the TOP level.

**f1 (Param Ramp):** Initiates a response profile for optimizing the following MS parameters: repeller, ion focus, entrance lens and X-ray. After the appropriate parameter is selected, the system ramps the voltage through the range selected in the Ramp Parameters EDIT form. The screen then displays the response profile for the indicated source element. At this time a new set of forms modes keys is available for the user. One of the keys is SET TO CURSOR in which the user can move the cursor to the desired voltage on the screen and set the voltage of the parameter. RETURN takes the user back to the preceding display. Pressing RETURN again takes the operator back to the EXECUTE keys.

**f2 (Profile Scan):** Provides a profile scan of the three masses entered in the Scan Params EDIT form.

**f3 (Repeat Profile):** Provides a repetitive profile scan of the three selected masses in real time. The operator can make parameter changes using the option table appearing on the right hand side of the screen display. Changes in tuning parameter values are accomplished by pressing the RETURN hardkey (on the keyboard), waiting for the ">" prompt in the upper right handcorner of the screen, and then typing (in capital or small letters) to increase or decrease the appropriate value and then watching the screen for changes in peak shape, resolution or abundance.

**f4 (Spectrum Scan):** Shows the mass spectrum obtained under current scanning and tuning conditions. Note that the spectrum is stored on disc. The name for the spectrum file is the default manual tune file name with the first character replaced with a ">". A directory is automatically created for the file. The file has no security code and therefore can only be purged by logging on with MANAGER.

**f5, f6 (not used)**

**f7 (Alt Options):** Moves the user to the alternate set of EXECUTE level keys which are:

**f1 (Width Calib):** Adjusts the Full Width at Half Max to 0.5 amu +/- .05 amu for the specified masses.

**f2 (Axis Calib):** Performs a mass axis calibration in order to precisely define the mass assignments over a particular range. The masses used are the three (or two) masses selected in the Scan Params EDIT form. For reliable results these masses should be at least 150 amu apart.

**f3 (Speed Tune):** Provides a repetitive display of the six selected masses in real time. The operator can make parameter changes using the option table appearing on the right hand side of the screen display. Changes in tuning parameter values are accomplished by pressing the RETURN hardkey (on the keyboard), waiting for the ">" prompt in the upper right hand corner of the screen, and then typing (in capital or small letters) to increase or decrease the appropriate value and then watching the screen for changes the abundance.

**f4 (MS Off):** Turns the mass spectrometer off.

**f5 (not used)**

**f6 (Edit):** Takes the operator to the EDIT level keys.

**f7 (Alt Options):** Moves the user to the alternate set of EXECUTE level keys.

**f8 (Top Level):** Takes the operator to the TOP level keys.

#### 3.1.2.4 Manual tuning procedure OPEN THE PFTBA VALVE.

Enter into the TOP level keys. If Autotuning was performed due to vastly altered conditions, hit Read Autotune. If not, hit New Tune File and enter the name of the normal tuning file. Tune file names are in the format TUNERn, where n is the number of the MS instrument. Proceed by hitting the Execute key.

When column connections have been changed or the vacuum system has been vented and restarted, look at a spectrum scan to observe if air or moisture is still present. Masses 28 and 32 are used to monitor air (although if a huge leak exists, apparent masses 14 and 16 could arise). Water is monitored with mass 18. If any of these are in large abundance, steps must be taken to eliminate the problem as these artifacts squelch sensitivity. The analyst can edit the target masses (through EDIT Tune Params) to 18, 28 and 32 and monitor these through Repeat Profile Scan.

If no artifacts are notably present, the tuning process can proceed normally. Look at the Profile Scan and note the peak area ratios, shapes, widths and masses. If the peak widths or positions need adjustments, perform these now through the Width Calib and Axis Calib processes and return to a profile scan. Correcting peak shapes is performed by trial-and-error through the Repeat Profile process of parameter changes. Good luck.

Since manual tuning is often performed because of a failure to satisfy BFB or DFTPP tuning criteria, record the relative abundances of the normally monitored masses (69, 131 and 219 for volatiles and 69, 219 and 502 for semivolatiles). Plan target abundances based on the failure of the BFB or DFTPP tune. **Keep in mind that the masses monitored with PFTBA are not the same as the EPA tuning compounds.**

Enter Param Ramp and view the ramp of the Repeller. All of the repeller maxima are normally always at 10.2 volts. Occasionally a high mass maximizes at a slightly lesser voltage, but if more than one mass shows this behavior or the ramps are virtually flat, there is a source configuration problem and chances are that the source must be removed and possibly cleaned. Other than these changes, the repeller is of no help in making alterations to relative mass abundances and does not have to be viewed again.

Next, view the X-ray ramp. All masses should maximize at the same voltage, which the parameter should be set at. The X-ray is of no help in altering the mass abundances and should not have to be viewed any more.

Ramp parameter responses are normally in the fifty thousand count range. Any ramp parameter showing a response of less than ten thousand counts indicates an source alignment problem or lack of PFTBA standard and should be corrected before proceeding.

The two parameters which greatly affect tuning are the Ion Focus and the Entrance Lens. **These parameters are very interdependent, as changes in one affect the response profile of the other.** View both without making any changes and notice the response patterns. The interrelationship of the responses of the low, middle and high masses should be carefully noted and used to adjust the relative abundances. Trial-and-error experience in this procedure enables the analyst to become proficient in manual tuning and reduce the time necessary to tune an instrument.

After setting the new ion focus and entrance lens parameters, perform Width and Axis calibration. Look at the Profile Scan and compare the abundances to the original targets. If the targets are close to the predicted needs, move to the TOP level keys and hit Store Tune. Enter the tuning file name for that instrument. **CLOSE THE PFTBA VALVE!**

Run a tune solution and compare these results to the previous tune. Make correlations between the changes in this result and the alterations performed during manual tuning. If this tune also fails criteria, return to MTUNE and repeat the process, using the comparisons of previous work to correct the parameters.

If minor changes are not helping the tuning procedure or if abundance ratio alterations are opposing, try making a gross adjustment to the ion focus or entrance lens. Be sure to move back and forth with changes to these parameters due to their interrelationship. If large changes are made, be sure to check the X-ray to maintain maximization.

It is desirable to set a parameter at a point which is relatively flat in the response profile. Parameters set in an area of rapid change (a high slope) tend to be more unstable. This is often not possible, especially when passable tune parameters are in a narrow window.

**CLOSE THE PFTBA VALVE!** Tuning is an art. Learn it.

### 3.2 Batch Data Acquisition

Batch monitoring and editing programs are used to setup and analyze groups of samples, including blanks and standards, automatically through sequence files.

#### 3.2.1 BEDIT - Batch Sequence Writer/Editor

Program BEDIT creates and edits batch sequence files which direct the acquisition of a set of samples. To run this program, type: BEDIT, with no parameters. BEDIT is a forms-driven program.

##### 3.2.1.1 BEDIT Namr form

This is the first form to appear in BEDIT and is the form BEDIT returns to after an edit is completed. Hitting the Exit key or entering a blank file name causes BEDIT to terminate.

The only data entered in this form is the namr of the sequence file to edit. This namr conforms to the standard FMGR format (name:sc:crn). The base format Kiber Analytical uses is in the form SEQ\_xx::SS. Type in this namr and hit the ENTER key. If the selected file does not exist, BEDIT displays a message asking the user if the file should be created. If the same namr is entered a second time, the file is created. Once a sequence file namr is successfully entered, the Header form appears.

#### 3.2.1.2 BEDIT Header form

The Header form is used to enter data that is global to the entire sequence file. The function of the f1 through f4 keys in this form are as follows:

**f1 (Sample Form):** This key must be used to jump from the Header form to the Sample form.

**f2 (not used)**

**f3 (Reseq Bottles):** This key causes the program to write sequential bottle numbers into any samples that has been written into the sequence. This is useful when a sample is inserted into the middle of a sequence.

**f4 (Zero Bottles):** not used in this procedure.

The fields entered in this form are as follows:

**Tune File Namr:** This is the namr of the tune file used for data acquisition, created by manual tune.

**Data File CRN's:** These fields define up to 10 cartridges to be used by BAMON. Input D1 and D3 as the first cartridges for VOA Instrument #1 and semivolatle Inst. #3. Cartridges D2 and D4 should be used initially for acquisitions from VOA Instrument #2 and semivolatle Instrument #4. When the number of free blocks on a cartridge falls below a user-specified number, the next cartridge in this list is used. The default for this list is the first cartridge in the user's cartridge list.

**Minimum Blocks for CR Check:** This number defines the number of blocks used as a "cut-off" point for switching cartridges. This number may be changed during acquisition through BAMON. Please note that if this number is less than the required space and the current cartridge fills up during an acquisition, the sequence pauses, effectively halting the run.

The remaining fields in this form are editing aids which affect how the Sample form operates.

**Start Edit at Compound Number:** When editing a sequence, the user may enter an existing sample number here, hit ENTER, then hit f1 (Sample Form) to jump directly to a specific sample.

**Use Autostep Mode:** If N is entered here, the user must use the "Next Sample" key in the Sample form to step to the next sample. If Y is entered here, the Sample form automatically steps to the next sample once all sample items are entered correctly and the user hits ENTER.

**Auto File Numbering:** If Y is entered here, the Sample form attempts to perform automatic file numbering. This feature uses the first data file name entered by the user and increment any numeric portion for the next file name.

**Use standard or alternate logic:** not used in this procedure.

#### 3.2.1.3 BEDIT Sample form

The Sample form is used to enter data for individual samples.

The function of the f1 through f4 keys in this form is as follows:

**f1 (Header Form):** This key causes the program to return to the Header form.

**f2 (Insert Sample):** This key causes the current sample and all samples following to be moved up one position allowing a sample to be inserted into a sequence.

**f3 (Previous Sample):** This key causes the Sample form to jump to the previous sample.

**f4 (Next Sample):** This key causes the Sample form to jump to the next sample.

The fields entered in this form are as follows:

**Data File Name:** the name of the data file used for the current sample. The first character is always a ">". To delete a sample from the sequence, blank out the data file name. When the file is created or replaced, all samples with blank names are removed.

**Name and Misc Data:** Enter the name and miscellaneous data. For samples, the Name entry consists of the laboratory's internal sample number (aannnnn) and the client or project Name. The Name entry for standards consists of the standard designation ISTDnnn [where I = V(olatile) or S(emivolatile) and nnn designates the concentration], the analysis date, and the instrument number. Blank sample Name entries are the same as standards except for the designation IBLKnnn, where nnn is a chronological descriptor for blanks. The Misc Data entry contains the client's sample number, the sampling date and time (if known), the fraction (if any), the dilution factor (if any), the instrument number and any other descriptive data. The defaults for these fields are the values entered for the previous sample.

**Method Namr:** Enter the namr of the acquisition method to be used for the sample. Note that samples in a sequence may use different methods.

**Bottle Number:** Enter the ALS bottle number for the sample on the semivolatile instruments. The default value for this field automatically increments as samples are entered. Note that the sample number and bottle number do not have to be the same. Enter -1 for manual injection. **Always** enter -1 for the volatile instruments.

**Sample Size:** Enter the ALS stroke, 1 to 5, which corresponds to approximately 1 to 5ul. Enter 1 for all narrow-bore capillary columns and 2 for wide-bore columns.

The following fields in this form deal with batch data processing for the sample. For further explanation, refer to the appropriate sections in this document.

**Quant:** Enter Y if batch quantitation is to be performed. This should be used except when analysis is for identification only.

**Report Format:** Enter N for None, S for Short, M for Medium, F for Full, E for Extended or P for Profile. Use N when not quantitating and M all other times. The other options can be separately performed after manual identification and integration operations.

**AuxReprt:** not used in this procedure.

**Quant ID File Namr:** Enter the namr of the Quant ID file to be used for batch quantitation.

**Quant Output File Namr:** Enter the namr of the Quant Output file to be produced by Quant. The defaults for this namr are:

Name: Same as data file name with ^ in place of the > symbol.

SC: (none)

CRN: The Aquarius default cartridge, SS.

Substitute V(olatile) or S(emivolatile) for ^ when additional quantitations through different ID files are performed

**Dilution Factor:** Enter the dilution factor to be applied to the sample in quantitation. The default for this field is 1.00. A negative number may be entered here to divide (instead of multiply) by this factor. Kiber Analytical virtually always uses the default as many post-acquisition operations require a base quantitation.

**Peak Label Method, Background Subtraction, Scale from n min., and n Page TIC** are not used in this procedure.

**Qion replaces Rt or Scan#:** Enter S to replace the Scan# column in Reprt with Quant Ion.

**Omit/Only (+/-) Types:** not used in this procedure.

**Mag Tape Archive:** Enter Y or N for archival of Data file, Quant ID file and/or Quant Output file. **NOTE: At the present time only the volatile instruments support automatic archival.** When all systems are in communication, the remaining instruments will utilize this feature. Until that time, **files are transferred from the non-archiving system to the archiving system by streaming tape and then archived manually through ARCV.**

The Data file is always archived. Once during each shift, after updating the calibration, archive the Quant ID file. Archival requests for the Data files and ID files are queued immediately after acquisition. Do not archive the Quant Output file at this time.

The remaining fields in this form deal with user programs and procedure files. Use of these is at the user's discretion.

### 3.2.2 BLIST - Batch Sequence List Program

Program BLIST produces listings of sequence files created by the BEDIT program. BLIST is a runstring-driven program that is used as follows: BLIST, seq file namr

### 3.2.3 BAMON - Batch Acquisition Control Program

The BAMON program allows monitoring and control of the batch data acquisition system. BAMON is a command-driven program which accepts commands to start, stop, pause and monitor the status of batch runs.

#### 3.2.3.1 Essential commands

Although the BAMON program accepts many commands to control the batch data acquisition system, the following steps are the bare minimum required to use the system:

- 1) Use the BA command to set up a sequence file for batch acquisition.
- 2) Use the GO command to start the run.
- 3) The ST command may be used to monitor the status of the run.
- 4) Use the EX command to exit BAMON.

Note that when the EX command is used to terminate BAMON, the batch run is **not** terminated. The batch data acquisition is in no way linked to the user's interactive session or terminal. The user may logon to any terminal on the system and re-run BAMON to check the run.

#### 3.2.3.2 Batch run states

When a batch run is set up on a given GC/MS system, it may be in any one of four states. These are as follows:

Ready - When a run has been set up by the BA command but has not yet started with the GO command.

Active - After the GO command, while a given sample is actually being acquired

Pause (User) - When a run is paused due to a user pause request.

Pause (Error) - When a run is paused due to an error (duplicate file name, no more disc space, etc.)

It should be noted that certain commands are only valid when the run is in a given state. This is discussed in the command descriptions section.

### 3.2.3.3 Summary of BAMON commands

**BA,system[,namr]:** Set-up single sample or sequence for batch run.  
**GO,system:** Start a run after BA command or resume paused run.  
**PA,system[,samp]:** Set run to pause after a given sample number.  
**MI,system[,num]:**  
    Display/change minimum number of blocks for CRN check.  
**SA,system,sample:** Set a paused run to a given sample number.  
**AB,system:** Abort run that is in the paused state.  
**ST,system:** Display complete status of an active system.  
**SY:** Display brief status of all GC/MS systems.  
**ER,system:** Display any error messages for a system.  
**AC,system[,access]:**  
    Display/change access to run (U=User, G=Group, S=System).  
**CS:** Run program CSPRM  
**LS,system:** Run program LSTGA (Show current RT, etc.).  
**SD:** Shut down the batch system (requires MANAGER logon).  
**SU:** Start up the batch system.  
**EX:** Exit BAMON.  
**?,command:** Detailed description of an individual command.

### 3.2.3.4 BAMON commands

The following is description of often used BAMON commands in detail. Other commands can be detailed by invoking the ? command or by consulting the Aquarius manual. NOTE: the GC/MS system number must be specified otherwise Instrument #1 is assumed.

**BA,system[,namr]:** The BA command sets up a batch run on the specified system and locks that system to the user's account. The run is then in the "ready" state. The default for system is GC/MS #1. Namr is the namr of the batch sequence file created by BEDIT. If this namr is omitted, BEDIT is invoked in the "single sample mode", which is not used in these procedures.

**GO,system:** The GO command starts a run that has been set up by the BA command or resume a run that is in the paused state. Note that checks for duplicate file name and free space on cartridges are performed at this time.

**PA,system[,sample]:** The PA command sets a pause flag on a given sample number in the sequence. When that sample is completed the run pauses instead of going to the next sample. To continue after a pause, use the SA command to call up the next sample, then the GO command to restart the run. If "OF" is used in place of the sample number the pause flag is cleared.

**SA,system,sample:** The SA command calls the sequence to a new sample number. The run must be in the paused or ready state to use this command. The default for system is GC/MS system #1. Sample number (required parameter) is the sample number (not bottle number) from the sequence file that the user wishes to call up.

**AB,system:** The AB command aborts the batch run on a given system. The sequence file is closed and the GC/MS system is unlocked. The run must be in the paused or ready state to use this command. If the user wishes to actually abort the current data acquisition, program CSPRM must be used. The default for system is GC/MS system #1. The batch monitor asks the user to confirm that the run is to be removed from the system. If Y is entered the run is aborted.

**ST,system:** The ST command displays in full the current status of a given GC/MS system. The default system is GC/MS #1.

**SY:** The SY command displays a brief status of all GC/MS systems currently configured in the user's data system including the identity of users currently owning GC/MS systems.

**ER,system:** The ER command displays the error log for a given system. If the specified system does not have a run set up on it, the error log is displayed for the last run completed on that system. The default for system is GC/MS system #1.

#### 3.2.3.5 Batch data acquisition sessions

When a run is set up using the BA command in BAMON, the user can notice a session logon message on the system console under the same User.Group. These 'non-interactive' sessions use session numbers 140 and down. The batch data acquisition runs under this session, **not** the user's terminal session. This is the means by which a user may logon/logoff without terminating acquisition. The batch session remains logged on until the end of the sequence is encountered (or the run is aborted with the AB command).

### 3.3 Quantitation Programs

This section describes the batch quantitation and batch reporting programs of Aquarius. It discusses the creating, editing and updating of Quant ID files as well as the use of the quant and quant report processes.

The Quant program uses "Quant ID files" that specify the criteria used to identify and quantitate target compounds. The raw data is analyzed using these criteria to produce a Quant Output file containing the results of the quant process.

Once produced, the Quant Output files may be used by other programs for such purposes as reporting and response factor calculation. In addition, user-written programs can make use of the information in these files.

#### 3.3.1 IEDIT - Quant ID File Writer/Editor

IEDIT is a forms-driven program that creates and edits ID files which direct the identification and quantitation of target compounds. To run this program type: IEDIT with no parameters. The following is a description of the forms and required data.

##### 3.3.1.1 IEDIT Namr form

This is the first form to appear in IEDIT. The program always returns to this form after an edit is completed. Hitting the Exit key or entering a blank file name at this point causes IEDIT to terminate.

The only data entered in this form is the namr of the ID file to edit. This namr conforms to the standard FMGR format (name:sc:crn). The base format Kiber Analytical uses is in the format ID\_xxx. Type in this namr and hit the ENTER key. If the selected file does not exist, IEDIT displays a message asking the user if the file should be created. If the same namr is entered a second time, the file is created. Once an ID file namr is successfully entered, the Header form appears.

Normal operations use a naming convention in which the above xxx=XYZ, where X is V (volatiles) or B (extractables), Y is C (initial calibration) or D (daily continuing calibration), and Z is the one digit instrument number. Examples include ID\_VC1 and ID\_BD4.

### 3.3.1.2 IEDIT Header form

The Header form is used to enter data that is global to the entire ID file. The function of the f1 through f4 keys in this form are as follows:

**f1 (Compound Form):** This key must be used to jump from the Header form to the Compound form.

**f2 (not used)**

**f3 (Generate RefNamrs):** This key allows the user to change the namrs of all Reference Spectrum files in the ID file to sequential file names. The program exits from forms mode and prompt for all required information.

**f4 (Change ALL Conc):** This key allows the user to change the concentration of ALL compounds (except ISTD's) in the ID file. The program temporarily exits forms mode and prompt for the required information.

The fields entered in this form are as follows:

**Title:** Enter a title (up to 60 characters) for the ID file.

**RT Window (+/- minutes):** Enter the time in minutes to be used as the RT window. The specified retention time for each compound +/- this value is the window in which the top of the peak must fall. Single peaks use 0.5 min. When peaks with spectral similarities are present in the same retention time region, a value down to 0.2 min. can be used, with caution, to eliminate erroneous "hits". Multicomponent targets may use broad RT windows.

**Maximum Hits per Compound:** not used in Kiber Analytical procedure due to the use of Auto QDel Method 5.

**Minimum area for Integration:** Enter 500, the minimum area counts to be detected by the data system. Later manipulation raises the final minimum area, but 500 permits some poorly integrated target peaks to be classified as a "hit".

**Peak/Base Peak Error Allowed:** Enter 50% error allowed in the relative abundance of masses to identify a compound. This extent allows a margin of variance for identification purposes apart from the final qualitative requirements.

**Slope Sensitivity:** Enter the slope sensitivity to be used by the data system integration. Entering a negative number here (-.2 instead of .2, for example) causes the integration to use a smoother slope calculation. Use .3 (sometimes .2 as determined by experimentation) as the entered value.

**Ignore Max Check (Y or N):** Enter a Y here to ignore the maximization check that is performed as part of the qualitative identification. The maximization check is **still performed**, however, peaks that fail are only flagged and are **not** rejected. Peaks that fail maximization check show the character ^ next to the area in all quant reports.

**Abundance Subtraction Method:** Enter the number 2 to calculate relative abundances by subtraction of the smaller of the first or last scan.

**Auto Qdel Method:** Enter 5 to select the best hit based upon the combined Q-value (quality of spectral match) and RT match.

**Units of Concentration:** Enter "ng/uL" for semivolatiles and "ug/L" for volatile analysis.

**Start Edit at Compound Number:** When editing an ID file, the user may enter an existing compound number here, hit ENTER, then hit f1 (Compound Form) to jump directly to a specific compound.

#### 3.3.1.3 IEDIT Compound form

The Compound form is used to enter the criteria for an individual compound. The function of the f1 through f5 keys in this form are as follows:

**f1 (Header Form):** This key causes the program to return to the Header form.

**f2 (Insert Compound):** This key causes the current compound and all compounds following to be moved up one position allowing a compound to be inserted into the ID file.

**f3 (Previous Compound):** This key causes the compound form to jump to the previous compound.

**f4 (Next Compound):** This key causes the compound form to jump to the next compound.

**f5 (Calib Form):** Go to the Calib(ration) form to allow edit of calibration information.

Compounds in the ID file need **not** be entered in retention time order. The order of compounds is used to determine how internal standards are used. When an ISTD compound is encountered by the Quant process, all compounds following it are quantitated based on that ISTD (until another ISTD is encountered). In addition, a single compound may be quantitated using more than one ISTD simply by entering it into the ID file in several different places. Note, however, that any given internal standard name must appear only once in the ID file.

The fields entered in this form are as follows:

**Compound Name:** Enter the name of the compound (up to 30 characters), using the nomenclature as designated in Labworks. Note: To delete a compound from the ID file, blank out the compound name. When the file is created or replaced all compounds with blank names are removed.

**Expected RT:** This field indicates the expected retention time for the compound. This may be entered in one of two ways:

- 1) A **positive** number indicates **relative** retention time.
- 2) A **negative** number indicates **absolute** retention time.

For internal standards, both positive and negative numbers are interpreted as absolute retention time. When the file is created or replaced, all absolute retention times for target compounds are converted to the calculated relative retention times.

**Qual Ions:** Enter up to five mass/relative abundance pairs to be used for compound identification (at **least** one must be entered). Note that the first mass is always 100% relative abundance (this number may not be changed in the form). Fractional masses (for example: 121.7) are allowed. NOTE: If relative abundance is entered as zero (or blank) that ion is for display only. Display only ions are **not** use for target compound identification but are displayed in the Reprt E and P formats.

The relative abundance values for a target compound should be comparable to the values found in a 20 ng/ul standard.

**Compound Type:** Enter an "S" for surrogate standards, but do not enter an "M" for matrix spike compounds. These conventions are needed to produce properly labelled ion chromatograms for CLP work. When entering new compounds into the ID file, the compound type is defaulted to I for internal standards and T for all targets.

**Peak Label Methods:** The five label methods are defaulted to N (Comp Name), R (R.T.), S (Scan#), # (Comp#) and T (Compound Type) when entering new compounds into the ID file. Enter a "U" into method number 5.

**User Label:** Enter the full compound name for all internal and surrogate standards. Leave blank for all other compounds.

**Ion for Quantitation:** Enter the ion to be used for quantitating the compound. The default for this field is the first (100%) mass entered above. This **must** be one of the five qualitative ions. Use the ion specified in the latest CLP Statement of Work or as listed in paragraph 7.2.3.1 of the volatile or semivolatile SOP's. If interferences with this ion are observed or expected make an additional, separate compound form entry for that target using the suggested alternative quantitation ion.

**Concentration of Compound:** Enter the concentration of the compound to be used for quantitation. This should be the concentration of the daily calibration check standard.

**Area Correction:** This is not normally used in Kiber Analytical procedures.

**Ref Spec Namr:** The entry into this field is performed by the QREF program. If this field is blank for a target compound which will be reported in full CLP deliverables, notify the GC/MS Supervisor or the Organics Group Leader.

**Internal Standard (Y or N):** Enter Y if this compound is to be used as an internal standard, N if it is not. The default for this field is Y for the first compound in the ID file and N for all others.

The remaining fields in this form are the same as the Quant parameters in the Header form. Enter these **only** if they are to be different from the global values in the header.

**NOTE:** The purpose of the ID file is to prescreen the Data files for target analytes. Parameters should be entered so that, if the target compound is present in the analysis, **it is not missed (causing a false negative)!** If the standard analysis indicates that **any** target fails to be found as a "hit", the ID file should be altered to produce the correct identification at all times. Many instances may arise that require liberal identification parameters to be used. False positives can be disallowed later in the process.

#### 3.3.1.4 IEDIT Calib form

The Calib form is used to edit the calibration information for a compound. Note that this information is normally placed in by calibration programs such as QCAL and CBCAL. This form allows the user to enter them manually. This process is **only** used when duplicating response factors for uncalibrated target isomers such as meta- and para-xylene and cis-1,2-dichloroethene.

The function of the f1 through f5 keys are as follows:

**f1 (Header Form):** This key causes the program to return to the Header form.

**f2 (not used)**

**f3 (Previous Compound):** This key causes the Calib form to jump to the previous compound.

**f4 (Next Compound):** This key causes the Calib form to jump to the next compound.

**f5 (Compound Form):** This key causes the program to return to the Compound form.

The fields in this form are as follows:

**Calib Method:** Enter 0 for single or average response factor, 1 for 1st degree curve or 2 for 2nd degree curve.

**RF:** Enter the response factor for the compound if using method 0. For methods 1 and 2 enter the appropriate factors in the equation.

**Rx and Ry:** This field is not used in Kiber Analytical procedures.

### 3.3.2 IDLST - Quant ID File List Program

Program IDLST produces listings of Quant ID files created by the IEDIT program. IDLST is a runstring-driven program that is run as follows: IDLST, ID file namr [,listlu [,M or F [,comp range]]]

**ID file namr** is the namr of the ID file to list.

**Listlu** is the device Lu to send the listing to. The default is Lu 1 (user's terminal).

**M** means Medium format listing (includes the mass/abundance information for each compound). **F** means Full format listing (shows ALL information in the ID file). If omitted, the short listing is generated.

**Comp range** is a compound number or range of compound numbers (comp1:comp2). The default is the entire ID file.

### 3.3.3 QT - Manual Quant Request

Program QT allows the user to issue a request to the Batch Quant Process. This request is "stacked" behind any other requests pending for Quant. Optionally, QT can run a local copy of Quant at the user's session, thus bypassing the Batch Quant request list. QT is a runstring-driven program that is used as follows: QT, Data namr, ID namr [,Output namr [,options [....]]].

The following is a description of the parameters used in Kiber Analytical procedures:

**Data namr:** The namr of the MS Data file (required parameter).

**ID name:** The namr of the Quant ID file (required parameter).

**Output namr:** The namr of the Quant Output file to be used. If it already exists it is over-written. The defaults for this are:

Name: Same as the data file name with ^ in place of > for the first character

SC: (none)

Crn: The Aquarius default cartridge, SS.

Note that there is no required naming convention for these files (as in MS Data files). Any valid 6 character file name is allowed.

**Options:** A combination of characters (not separated by commas). Kiber Analytical operations use the following options applying to QUANT:

- !** - Run now (run copy of quant at user session). Use **!!** to echo at user terminal.
- R** - Run QREP after QUANT execution. Only valid with the **!** option.
- T** - Test mode (output trace of QUANT process). This option is very useful in determining what factor(s) causes a target to be missed.
- {range}** - Specify **{comp#}** or **{comp#:comp#}** to use only part of the ID file. The braces **{}** are required.

The following options applying to REPRT are used in Kiber Analytical procedures:

- S, M or E** - Request REPRT with **Short, Medium or Extended** format (default is **no report**). **M** is the standard option at Kiber Analytical and generates a chromatogram with the Quant Output file. Option **E** is **only** used for an extended look at a PE (performance evaluation) sample or when requested by the Organic Group Leader, the QC/QA Coordinator or the Division/Laboratory Manager.
- Q** - Replace the RT column in the tabular report with Quant Ion.  
Use **QQ** to replace the Scan# column instead. **Use of option QQ is mandatory at Kiber Analytical.**

### 3.3.4 QREP - Tabular Quant Report

Program QREP produces a quick tabular report from a Quant Output file without going through the REPRT process. QREP is a runstring-driven program that is used as follows: QREP, Quant Output file namr [,listlu [,options]]. The default listlu is lu 1 (user's terminal). The report produced by this program is identical to the tabular (short) report produced by REPRT. Options are as follows:

- N** - Show compounds no found (no hits).
- D** - Show compounds that have been deleted (see program QDEL).
- Q** - Replace the RT column of the report with Quant Ion.
- QQ** - Replace the Scan# column of the report with Quant Ion.

A hardcopy of the final Quant Output file in tabular format is generated for each quantitated analysis using the QQ option.

### 3.3.5 QDEL - Quant Output File Record Deletion Program

Program QDEL allows the user to delete records from a Quant output file and eliminate false and multiple hits. The user is prompted for the correct hit and all others are deleted. QDEL is a runstring-driven program that is used as follows: QDEL, Quant Output file [,option [,start comp #]]. Option may be one of:

- A** - Ask for correct hit even for single hits and internal standards (default mode is to ask only for multiple target hits).
- E** - Edit mode. In this mode, instead of selecting one correct hit, the user is prompted for each peak in the output file. Internal standards and single hits are included in edit mode.
- U** - Undelete. This option clears all delete flags, thus undeleting all hits in the file.
- Q** - Similar to U, but file is returned to original Quant conditions. Undelete all hits, then delete hits that were automatically Qdel'ed by Quant.

**Start comp #** - optionally allows the user to start at any compound in the output file. This parameter is ignored with the U and Q options. Records that are deleted with QDEL are only **flagged** as deleted (not actually removed from the file). These records are then ignored by all other Aquarius programs (except for QREP using the D option).

QDEL is used in Kiber Analytical procedures primarily to remove false positive hits. Since Kiber Analytical utilizes Method 5 to select the best hits, multiple hits are not produced. Therefore Kiber Analytical uses QDEL with option A or Q only.

### 3.3.6 QREF - Save Reference Spectra

Program QREF reads a standard Quant Output file, extracts a background subtracted spectrum for each compound from the MS Data file and saves the spectrum under the name specified in "Ref Spec Namr" in IEDIT (of the ID file used to produce the output file). QREF is a runstring-driven program that is used as follows: QREF, Quant Output file namr [, options [, start comp #]]. Options may be any of the following letters:

- A - Ask for correct hit even for single hits and internal standards.
- Y - Automatic YES option. This tells QREF that it is OK to SAV or reSAV the spectra. If this option is **not** used, QREF draws each spectrum and request confirmation before the SAV is executed.
- N - This option is not used in Kiber Analytical procedures.

**Start comp #** - allows the user to specify a compound number in the output file to start at. The default is compound number 1.

Most target spectra have been previously saved. Some target spectra are difficult to acquire and may take considerable effort to regenerate. Therefore **the analyst should overwrite a reference spectrum only if permission from the GC/MS Supervisor and the Organics Group Leader is granted.** If this program is used to generate reference spectra, choose between **any** MS Data file to find the best example of a "clean" spectrum.

### 3.3.7 QAREA - Quant Manual Integration Program

Program QAREA allows the user to display extracted ion profiles for the hits in a Quant Output file and optionally re-integrate them using the graphics cursor. Qarea is used as follows:  
QAREA, Quant Output file namr.

Once QAREA is started, it accepts simple 1 and 2 character commands. The commands are listed on each screen and are self explanatory.

The key command, I (manually integrate using cursor), should be used judiciously and correctly. Cursor settings for foreground and backgrounds should account for all baseline aberrations. The full area of the target peak, including tailings, should be integrated. Interferences which skew integrals should be noted and compensated for, if possible. If interferences cannot be removed by integration, the alternate quantitation ions should be used. No peak slicing, cutting, truncating is allowed. No additional peaks can be integrated with the target peak (except on multicomponent targets).

Once a peak has been integrated with the QAREA I command, all Quant reports print an M next to that peak's area. Note that no changes are actually made to the Quant Output file until the E command (exit and replace output file) is used to exit. If changes are made to the file, Qarea asks the user if the resultant Quant Output file should be automatically archived. **If this is a finished and final output file (including any QDEL operations), archive it now.** This is the only archiving (except possibly manually) performed on output files.

**NOTE: At the present time only the volatile instruments support automatic archival.** When all systems are in communication, the remaining instruments will utilize this feature. Until that time, files are transferred from the non-archiving system to the archiving system by streaming tape and then archived manually through ARCV.

### 3.3.8 QRP - Manual Report Request

Program QRP allows the user to issue a request to the REPRT process to produce a Quant report. This request is "stacked" behind any other requests pending for REPRT. QRP is a runstring-driven program that is used as follows: QRP, Quant Output file [,options [Aux Reprt [,scale time]]].

Options may include one of the following formats:

- S** - Short format: A tabular report of the identified target compounds.
- M** - Medium format: The tabular report plus a total ion chromatogram.
- F** - Full format: The tabular report, the total ion chromatogram and a mass spectrum for each of the identified target compounds.
- E** - Extended format: Same as Full with the addition of extracted ion profiles for each of the ions used in quantitation.
- P** - Profile format: The tabular report, the total ion chromatogram and a page for each compound. This page contains the extracted ion profiles for each ion over the entire compound window and a list of all the hits.

Plus any of the following additional options:

- L** - Peak label option. A method (1-5; default=1) may follow the L.
- B** - Shows background subtracted spectra (only applies to E and F formats).
- C** - Tells Reprt to cut the total ion chromatogram (TIC) into multiple pages. The number of pages to use (1-99; default=2) may follow the C. The TIC is divided (by retention time) into the number of pages requested. The retention time range of each page overlaps the surrounding pages by 2% (in case a peak is split between pages).
- Q** - Replace the RT column of the tabular report with Quant Ion. Use QQ to replace the Scan# column instead.

- - (minus sign) is not used in Kiber Analytical procedures.

+ - (plus sign) is not used in Kiber Analytical procedures.

{range} - Specify {comp#} or {comp#:comp#} to report graphics on a single compound or range of compounds. This applies to the Full, Extended and Profile reports.

**Aux Reprt:** not used in Kiber Analytical procedures.

**Scale time:** (in minutes) tells Reprt where to start scanning the data file when determining the largest scan. The largest scan found after this point is used to scale the total ion chromatogram. This is used to prevent a large solvent peak from causing poor scaling in the TIC. The default value is 0.00 (scale to the largest run in the scan).

### 3.4 Calibration Programs

This section describes the single-point and multi-level calibration capabilities of Aquarius. Single-point (daily or continuing) calibration is performed by the QCAL program. The multi-level calibration feature allows the use of up to 10 data points in the calibration of an ID file. This calibration may be performed on a compound by compound basis using an average response factor or a curve. This feature makes use of an intermediate "Calibration file" which stores response ratios and concentrations. The programs described in this section allow the user to create, edit, update and otherwise manipulate these files.

#### 3.4.1 QCAL - Quant ID File Calibration Program

Program QCAL uses a Quant Output file to calculate response factors and update a Quant ID file. Qcal is a runstring-driven program that is used in as follows: Qcal, Quant Output file [,ID file [,options]]. The following is a description of these parameters:

**Quant Output file:** The namr of he Quant Output file to use in response factor calculation (required parameter).

**ID file:** The namr of the Quant ID file to place response factors into. If omitted, the ID file that was used to create the Quant Output file is updated.

**Options:** A combination of the following letters (not separated by commas):

- C** - Clear all previous calibration from the file before updating. If this option is not used, previous calibration is retained for compounds that are not updated.
- A** - Ask mode. The program asks for confirmation on all compounds, allowing the user to avoid unwanted updates. If this option is not used, QCAL only asks in the case of multiple hits.
- T** - Update retention times **only**. Response factors are not altered.

The calibration time stamp in the ID file is updated by this program.

#### 3.4.2 CBCRE - Create Calibration File from ID File

Program CBCRE creates a new Calibration file using the compounds in an existing Quant ID file of the format ID\_xCx. CBCRE is a runstring-driven program that is used as follows: CBCRE, ID file namr, Calibration file namr, where ID file namr is the namr of an existing Quant ID file and Calibration file namr is the namr of the new Calibration file to create. Both parameters are mandatory.

The new file is created and the following information is written into it:

- 1) The 60 character title is the same as the ID File title.
- 2) The units of concentration are the same as in the ID file.
- 3) All target compounds are taken from the ID file paired up with the appropriate ISTD name.
- 4) The file is defaulted to 5 concentrations (10, 20, 50, 100, 200) and a check standard concentration (100).

Once the file has been created, any of the above items may be edited using the CBEDT program.

### 3.4.3 CBEDT - Calibration File Writer/Editor

CBEDT allows the user to create/edit Calibration files. To use, type: CBEDT with no parameters. CBEDT is a forms-driven program. The following is a description of the CBEDT forms and required data.

#### 3.4.3.1 CBEDT Namr form

This is the first form to appear in CBEDT. The program always returns to this form after an edit is completed. Hitting the Exit key or entering a blank file name at this point causes CBEDT to terminate.

The only data entered in this form is the namr of the calibration file to edit. This namr conforms to the standard FMGR format (name:sc:crn). The naming format for volatiles is "VOAimx" where **i** = Instrument number, **m** = matrix [**S**(oil) or **W**(ater)] and **x** = the designation of the calibration (chronologically **numeric** for operations under the 1988 CLP SOW and **alphabetic** for calibrations under the 1990 CLP SOW). Semivolatile calibration namrs follow the format "CAiaax" where **i** = Instrument number, **aa** = a calibration usage code (BC is base calibration, DL is for a EPA detection limit study, and HV is for a Hubaux and Vos detection limit study), and **x** = the alphabetic designation of the calibration. Type in this namr and hit the ENTER key. If the selected file does not exist, CBEDT displays a message asking the user if the file should be created. If the same namr is entered a second time, the file is created. Once a sequence file namr is successfully entered, the Header form appears.

#### 3.4.3.2 CBEDT Header form

The Header form is used to enter data that is global to the entire calibration file. The function of the **f1** through **f4** keys in this form are as follows:

**f1 (Compound Form)**: This key causes the Compound Form to appear.

**f2 (IFB Form)**: This key causes the IFB form to appear (see note in the Title field below).

**f3 (not used)**

**f4 (not used)**

The fields entered in this form are as follows:

**Title:** Always enter "IFB" to set up the file for IFB format reporting. This allows automatic criteria checking for comparison of proposed continuing calibrations to initial calibrations

**Units of Conc:** Should be the same as in the ID file. This is for reporting purposes only (it has no effect on the calculation).

**Conc#1-10:** For each of up to ten data points, enter the concentration of each point. The number of entries here define how many data points are used in the calibration file.

**Check Std Amount:** Enter the concentration of the check standard. This is used only by the check standard report program (CBCHK).

**Start Edit at Compound Number:** When editing a Calibration file, the user may enter an existing compound number here, hit ENTER, then hit f1 (Compound Form) to jump directly to a specific compound.

#### 3.4.3.3 CBEDT IFB form

The IFB form is used to enter the data that appears on the IFB format calibration reports and check standard reports. The function of the f1 through f4 keys are as follows:

**f1 (Compound Form):** This key causes the Compound Form to appear.

**f2 (Header Form):** This key causes the Header form to appear.

**f3 and f4 (not used)**

The fields in this form are self-explanatory and should be filled out exactly as they should appear on the reports (no checking is done on these fields). Enter the required calibration criteria (Minimum RF, %RSD and %Diff) for the particular methodology.

#### 3.4.3.4 CBEDT Compound form

The Compound form is used to enter the information for a single compound in the Calibration file. The function of the f1 through f5 keys in this form are as follows:

**f1 (Header Form):** This key causes the program to return to the Header form.

**f2 (Insert Compound):** This key causes the current compound and all compounds following to be moved up one position allowing a compound to be inserted into the Calibration file.

**f3 (Previous Compound):** This key causes the compound form to step to the previous compound.

**f4 (Next Compound):** This key causes the compound form to step to the next compound.

**f5 (R.F. Form):** This key causes the program to go to the Response Factor form for the current compound.

The fields entered in this form are as follows:

**Comp Name and Istd Name:** Enter the name of the compound and the name of the appropriate Istd into these fields (leave Istd name blank for external standard compounds). **Note** that these names must **exactly** match the names in the ID file for proper identification. As an alternative to typing these names in, the names may be extracted from an existing Quant ID file (see next field). To delete a compound from the Calibration file, blank out the Comp Name.

**ID file and Comp#:** To avoid typing the above compound names in long hand, enter the namr and compound number of a compound in an existing Quant ID file here. The compound name and Istd name are extracted and placed into the Comp Name and Istd Name fields.

**Rx and Ry:** These are not used in Kiber Analytical procedures.

**Conc#1-10:** Only concentrations that are different from the value in the Header are entered here.

**Check Std Conc:** Only enter a check standard concentration if it is different from that in the Header.

**CCC and SPCC:** Enter Y or N for each of these fields. These identify which compounds are Calibration Check Compounds and System Performance Check Compounds. These apply only to IFB format reports.

#### 3.4.3.5 CBEDT RF form

The RF form allows the user to edit the response factor information for a compound. This information is normally placed into the file by CBUPD but may be entered or edited manually here. For each compound there is one set of parameters in the RF form for each concentration level. The function of the f1 through f5 keys are as follows:

**f1 (Header Form):** This key causes the program to return to the Header form.

**f2 (not used)**

**f3 (Previous Conc):** This key causes the RF form to step to the previous concentration (data point) for this compound.

**f4 (Next Conc):** This key causes the RF form to step to the next concentration (data point) for this compound.

**f5 (Compound Form):** This key causes the program to return to the Compound form for the current compound.

The fields in this form are as follows:

**Relative Retention Time:** Enter the value for (Std RT/Istd RT) for this data point.

**Response Ratio:** Enter the value for (Area Std/Area Istd) for this data point. Note that this is the Rm value.

**Conc of Istd:** Enter the Istd concentration for this data point. This number is normally taken directly from the Quant output file by program CBUPD (Istd concentrations are the only concentrations taken from the Quant output files).

**Finished RF:** (Optional) If a finished response factor is entered here, the response ratio (Rm) is recalculated automatically.

Note that Istd conc and Istd area are 1.0 for external standard compounds.

#### 3.4.4 CBUPD - Calibration File Update Program

Program CBUPD uses Quant output files to calculate response ratios and places them into the Calibration file. CBUPD is a runstring-driven program that is used as follows: CBUPD, Calibration file namr [,options]. Options are one or more of the following:

- C** - Clear previous calibration from the Calibration file first.
- A** - Ask for correct hit always. Allows the user to avoid update on a data point. Otherwise the program asks only in the event of multiple hits or if a data point is already present in the Calibration file.

CBUPD prompts for a Quant Output file namr for each of the concentrations in the Calibration file. To skip any of the concentrations, hit RETURN. This program may be run over and over until the calibration information is complete.

#### 3.4.5 CBRPT - Calibration Report Program

Program CBRPT produces calibration reports from the calibration file. Unfortunately, the output does not fit Kiber Analytical printer modes. Use, instead, a soft-key listing of the calibration file through the Utility Programs:AQUARIUS BatchQNT :Aquarius Listers:Cal file Lister pathway (or by the runstring order ",AQ1203"). This mini-program compresses the print to produce a readable report.

#### 3.4.6 CBCAL - Recalibrate ID File from Calibration File

Program CBCAL uses the information in the Calibration file to update an existing Quant ID file for use by the Quant program. CBCAL is a runstring-driven program that is used as follows: CBCAL, Calibration file namr, ID File namr [,comp# [,cal meth]].

The Calibration file and ID file must already exist. All data points in the Calibration need not be present; the calibration is calculated on however many points are present for a compound. **Comp#** is an optional compound number in the Calibration file to start calibration at ( the default is 1). **Cal meth** can be supplied to tell CBCAL which calibration method to use (A = average rf; 1 = 1st degree; 2 = 2nd degree). Note: If the requested curve cannot be used for a compound (not enough points or no unique solution), CBCAL drops to the next lower method (from 2nd to 1st and finally to an average rf).

If cal meth is omitted from the runstring, selection is made compound by compound. For each compound, the program displays such information as response factors, %RSD and correlation coefficients. The user must then enter one of the following options:

- A** - Use an average response factor.
- 1** - Use a 1st degree curve.
- 2** - Use a 2nd degree curve.
- P** - Retain the previous calibration in the ID file (this is displayed for each compound).
- E** - Exit program CBCAL (the user may continue later by re-running CBCAL using the optional comp#).

For any given compound, if CBCAL shows the message "Not enough data points" or "No unique solution" under 1st or 2nd degree curve, then these are not valid options for that compound.

#### 3.4.7 CBCHK - Calibration Check Report Program

Program CBCHK uses a Calibration file and Quant output file(s) to produce a calibration check report from a continuing standard injection. CBCHK is a runstring-driven program that is used as follows: CBCHK, Calibration file namr. Calibration file namr is the namr of an existing Calibration file that contains valid calibration information.

Once the runstring is entered, CBCHK prompts for Quant output files over and over again until the user hits RETURN. The report is automatically produced at the user's terminal printer.

### 3.5 Archiving Programs

#### 3.5.1 Archival Operations

All data files are archived twice, once with automatic archiving through Aquarius on 9-track tape and once on streaming tape. Streaming tape archiving is performed at least once each month. Procedures for this are found in the Utilities Manual of RTE-A. Aquarius archiving is done automatically through input in BEDIT, when exiting QAREA, or manually by ARCV. When manual 9-track archiving is performed, use the ARCV utility. Directory files for each tape are kept in a discrete notebook.

### 3.5.2 ARCV - Manual Archive Request

Program ARCV allows the user to manually issue requests to the archive system. Although automatic archival in the Batch Data Acquisition software is limited to Data, ID and Quant Output files, the ARCV program may be used to archive **any** file type.

ARCV is a runstring-driven program used as follows: ARCV, namr (single file) or ARCV, namr mask (multiple files). The requested file(s) is queued to the archive program. The namr mask uses "-" as the wild card character. All archive requests are "stacked" behind any other requests waiting for ARCH.

When a type 10 MS data file (>) is archived, the type 2 directory file (<) is automatically archived by the archive program. Therefore, the user need not request archive on these files.

### 3.5.3 Auxiliary archiving programs

Streaming tape archiving is performed using FC or FST program modes. Aquarius archival programs for setup (ARINI) and file access (ARUTL and ARQU) facilitate directory and file retrieval operations. Instructions for these programs can be found in the RTE-A Utility Manual (FC and FST) and in the Aquarius section of the main data system manual (ARUTL and ARQU).

## 3.6 Method Program Files

### 3.6.1 Method File Creation

Method creation is started by entering the soft key Create Method under Data Acquire, or by typing: ,\*TASK,15. When prompted, enter the number of the instrument. After the GC/MS instrument has been selected, a series of forms is presented sequentially.

#### 3.6.1.1 Sample information form

This form requests specific information for acquisition. During method file creation for batch operating, this form only needs only slight alteration. Be certain that the **Review input** field is filled with a "Y". After filling other fields with other "dummy" information, if necessary, press the **Enter Data** softkey.

#### 3.6.1.2 Temperature zones form

Enter the desired temperatures (paragraph 4.0 of the volatile or semivolatile operating procedures) for the injector port and interface.

#### 3.6.1.3 GC/DIP parameter table form

Enter the desired parameters to control temperature programming.

#### 3.6.1.4 Run time events form

The **Run Time** is calculated from the GC parameters in the previous form. This time may be shortened to stop acquisition before the GC run is over, but this is not recommended unless special circumstances warrant. The **Scan Start Time** is used to delay the filament operation and mass spectrometer scanning until unwanted components (solvent, etc.) have eluted from the gas chromatographic column. Volatile scanning can start immediately but semivolatile scanning should be started at 4 min after injection for methylene chloride or up to 10 min for later eluting solvents. The **Splitless Valve Time** should be set (on semivolatile instruments only) at 0.6 to 0.8 minutes. The **Relay Operations** are only used for purge and trap operations and should not be changed.

#### 3.6.1.5 Scan parameter form

The only fields normally modified on this form are the **Scan Range** and **Multiplier Voltage**. The scan range should be set according to paragraph 4.0 in the particular SOP but can be expanded for special targets. The multiplier voltage is usually altered after tuning procedures indicate a change in sensitivity.

#### 3.6.1.6 Method saving and creation

Whereas a previous method file was used to review parameters and make changes, this final screen allows the creation of a new method file. Select "Yes" to save the experimental method and create a new method by entering a new namr and designating SS as the cartridge.

#### 3.6.2 Method File Listing

Use the command: LMETH to obtain a listing of a particular method file. Log the screen to create a hardcopy printout.

#### 3.7 Directory File Reconstruction

The program FIXDA is used to regenerate corrupted data file directories. Type: FIXDA and then enter the namr and cartridge of the corrupt data file. The reconstructed files must be designated with a new namr of cartridge.

### **3.8 CLP Forms Generation**

Operations to generate CLP data deliverables in hard copy can utilize the soft-key programs supplied with Aquarius. These operations may also be performed by public domain software as shown in the section titled Automated Data Reporting/Generation.

At the current time we utilize public domain software. The backup procedures for forms generation in Aquarius will be delineated in this section when time permits.

## GC/MS VOLATILES ANALYSIS

### 1.0 SUMMARY OF PROCEDURE

This procedure is used for the GC/MS analysis of volatile samples after GC screening. Helium is bubbled through a sample contained in a glass purging chamber. The purgeables are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with helium to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer. The laboratory where volatile analysis is performed should be completely free of solvents, except for reagent methanol.

### 2.0 APPARATUS, REAGENTS AND SUPPLIES

#### 2.1 Apparatus

##### 2.1.1 Balances

An analytical balance capable of accurately weighing  $\pm 0.0001$  g is used for preparing standards. Soil samples are measured on a top-loading balance capable of weighing  $\pm 0.1$  g.

##### 2.1.2 Ovens

A small oven capable of heating to  $105^{\circ}$  C is utilized as a sparge glassware drying oven and also employed in moisture determinations.

##### 2.1.3 Purge and Trap Device

The purge and trap system (P&T) consists of a 16-place automatic sample purger, a trap/desorption unit and a sample heating unit.

##### 2.1.3.1 Purging chambers

Aqueous samples are purged through 5 mL or 25 mL medium porosity fritted glass spargers. The 5 mL spargers are used except for Method 524.2. Mixed phase samples (solids w/reagent water) are purged in 25 mL needle spargers at elevated temperatures.

#### 2.1.3.2 Trap

The trap is at least 25 cm long and has an ID of at least 0.105 inch. The trap is packed with at least 15 cm of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh), 8 cm of silica gel (Davison Chemical, 35/60 mesh, grade 15) and 1 cm of OV-1 (Ohio Valley, 3% on 60/80 mesh Chromosorb-W).

#### 2.1.3.3 Desorption unit

The desorption unit includes a water management system (O.I. Revision F or later) and an optional anhydrator. The anhydrator (used when a GC/MS system does not use a jet separator) must be preconditioned with acetone or other highly volatile ketone to equilibrate it, allowing analysis of target ketones.

#### 2.1.3.4 Temperatures

Water samples and medium-level soil samples are purged at ambient temperature. Low-level soil samples are purged at 40° C (+/- 1° C). These conditions are followed except when analyzing target compounds which require alternative sample heating conditions. The trap is rapidly pre-heated to 160° C immediately prior to desorbing at 180° C. The desorption unit valve, the transfer line between the automatic sampler and the desorption unit, and the transfer line between the desorption unit and the gas chromatograph are at 80° C during analysis.

#### 2.1.4 GC/MS System

The GC/MS system consists of a gas chromatograph, analytical column, mass spectrometer, data system and archival system.

##### 2.1.4.1 Gas chromatograph

The GC system must be capable of temperature programming and have flow control that maintains a constant column flow rate during desorption and temperature program operations. The transfer line from the P&T desorption unit is either be interfaced to a low dead-volume injector port or directly connected to the analytical column. All GC gas carrier lines must be constructed from solvent-cleaned stainless steel tubing, thoroughly dried before utilization. Non-PTFE P&T tubing, non-PTFE thread sealants, or flow controllers with rubber components are not used. The column oven must have the capability of subambient operation.

##### 2.1.4.2 Analytical column

30 m long x 0.53 mm ID DB-624 megabore fused silica capillary column with a 3 um film thickness or a 30 m long x 0.32 mm ID DB-624 widebore fused silica capillary column with a film thickness of 1.8 um is used. The column inlet is connected to a low dead-

volume injector port if the P&T transfer line is also connected. When the P&T transfer line is interfaced directly to the capillary column a stainless steel zero dead-volume union (J & W Scientific) with specific capacities (1/16" to 0.53 mm or 1/16" to 0.32mm) is used. When utilizing a widebore column, the column outlet is directly interfaced to the mass spectrometer so that the end of the column resides 2 mm from the source repeller. Operating with a megabore column requires utilization of a jet separator interface between the column and the mass spectrometer.

#### 2.1.4.3 Mass spectrometer

A quadrupole mass spectrometer utilizing 70 volts (nominal) electron energy in the electron impact ionization mode is used. It must be capable of scanning from 35 to 300 amu every 3 seconds or less and capable of producing a mass spectrum which meets the appropriate tuning criteria when 50 ng of 4-bromo- fluorobenzene (BFB) is purged through the analytical system. The instrument pumps has a carbon trap to collect effluents and must be vented to the outside of the facility to prevent the release of untrapped contaminants into the instrument room.

#### 2.1.4.4 Data system

A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has Aquarius software which allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This software allows integration of the abundance of any such plot between specified time or scan number limits. This software allows the comparison of sample spectra of unknowns against reference library spectra in the 1989 release of the NIST/EPA/MSDC mass spectral library. The data system is capable of flagging all data files that have been edited manually by laboratory personnel.

#### 2.1.4.5 Archival system

A 9-track magnetic tape storage device must be available to record data and each data system must have a streaming tape device. All must be suitable for long-term, off-line storage, at least one storage area segregated from the GC/MS laboratory.

#### 2.1.5 Power Backup and Conditioner

An uninterruptable power supply system is used which can supply full power to the purge and trap device and the complete GC/MS system, for at least 30 minutes, in the event of a power failure. This system provides power line conditioning to prevent

disturbances due to surges, spikes and droppages.

## 2.2 Reagents and Supplies

### 2.2.1 Reagents

#### 2.2.1.1 Reagent water

Reagent water is defined as water in which an interferent is not observed at or above the detection/quantitation limits. Reagent water may be purchased as commercial bottled water or generated in house from deionized water which is further purified by a MILLI-Q water system, boiled for 15 minutes and then, while maintaining the temperature at 90° C, bubbling contaminant free N<sub>2</sub> through the water for one hour. **Containers of reagent water are KEPT CLOSED AT ALL TIMES, except when dispensing water.**

#### 2.2.1.2 Blank soil

Soil free of significant organic compounds is heated to 400° C overnight. Sand should not be used as blank soil due to the large difference in adsorption characteristics in relation to most soils. **Containers of blank soil are KEPT CLOSED at all times, except when dispensing.**

#### 2.2.1.3 Methanol

The methanol used for GC/MS volatile analysis is purge and trap grade. Commercially supplied reagent bottles must be no larger than 250 mls to minimize accumulation of contaminants.

#### 2.2.1.4 Gas and cryogenic liquid supplies

The carrier/purge gas is zero grade helium supplied through a two-tank manifold which allows tank removal without disruption of the supply. The gas is equipped with an in-line trap for the removal of moisture and oxygen, with a hydrocarbon trap within the P&T system. Compressed air is supplied for solenoid valve operations. Liquid nitrogen is used for cryogenic GC oven operation. The supply line can be insulated copper but vacuum glass lines are preferred.

### 2.2.2 Supplies

#### 2.2.2.1 Syringes

##### 2.2.2.1.1 Sample introduction

A 5 mL or 25 mL gas-tight "Luerlock" syringe with miniature inlet valve is used for introduction of the sample into the sparge tube.

#### 2.2.2.1.2 Aliquot introduction

Various sized gas-tight microsyringes with built-in shut-off valves are used to deliver aliquots of sample or methanol extract (medium level soil analysis). If a dilution of 2500X or larger is needed, a 10 uL microsyringe may be carefully employed.

#### 2.2.2.2 Glassware

##### 2.2.2.2.1 Volumetric glassware

Clean class A volumetric glassware at ambient temperature is used for dilutions.

##### 2.2.2.2.2 40 mL VOA vials

EPA Class II pre-cleaned 40 mL VOA vials are used in medium level soil analyses.

##### 2.2.2.2.3 4 oz. wide-mouth jars

EPA Class II pre-cleaned 4 oz. wide-mouth jars are used to store blank soil.

##### 2.2.2.2.4 Disposable pasteur pipettes

##### 2.2.2.2.5 Automatic sampler vials, GC

#### 2.2.2.3 Dessicator

A dessicator with an indicating drying agent is used in moisture determinations.

#### 2.2.2.4 Weighing Dishes

Aluminum weighing dishes, 8 mm deep x 60 mm diameter, are used for moisture determinations.

### 3.0 STORAGE AND PREPARATION

#### 3.1 Sample Storage

##### 3.1.1 Sample Custody

VOA samples are transferred from the Sample Custodian to the volatiles analyst at the entrance of the GC/MS laboratory, accompanied by the internal analytical request form, chain of custody and any related paperwork. Samples using full Contract Laboratory Program (CLP) protocol are then signed out for GC/MS VOA/Storage on the associated Organic Checkout/ Checkin Form by the volatiles analyst, who is responsible for the custody of those samples until the analyses have been completed, the final

report received by the client, and the leftover sample/duplicates have been returned to the Sample Custodian and signed back in on the Organic Checkout/Checkin Form.

### 3.1.2 Sample Storage

All volatile samples are stored in a discrete VOA refrigerator at 4°C (+/- 2° C) located in the GC/MS laboratory. The only exception to this rule occurs when gross contamination is detected by olifactory sensation or screening procedures, in which case the samples remain in the central storage refrigerator in the sample receiving department. VOA sample vials are kept sealed with TFE-lined caps in appropriate sample racks to prevent alteration, contamination, excess movement and breakage. The actual refrigerator temperature is monitored and recorded every workday. The refrigerator is kept locked when not attended by the volatiles analyst(s) for any extended period. The volatiles analyst(s) and the GC/MS supervisor are the only persons in possession of the keys to the GC/MS VOA refrigerator.

### 3.2 Analytical Standard Storage

All analytical standards in solution are stored with minimal headspace in screw-cap micro-reaction vessels equipped with Mininert valves in the closed position. These solutions along with neat standards are stored in a discrete VOA freezer at -10° C to -20° C located in the GC/MS laboratory. Alternative storage methods can be used if the above storage conditions are known to be detrimental to the integrity of a standard or unsafe. Each standard is discretely designated and chronologically entered into a bound GC/MS Standards Notebook along with all preparation operations and/or manufacturer's documentation. Quarterly inventories of standards are made and recorded in this notebook.

### 3.3 Analytical Standard Preparation

#### 3.3.1 Target Calibration Standards

##### 3.3.1.1 Stock standard solutions

Stock standard solutions may be prepared from pure standard materials or purchased. Prepare stock standard solutions in methanol using assayed liquids. In-house preparation of stock standards from gases is not recommended.

Place about 9.8 mL of methanol into a 10.0 mL tared ground glass stoppered Class A volumetric flask. Allow the unstoppered flask to stand for about 10 minutes or until all the alcohol wetted

surfaces have dried. Weigh the flask to the nearest 0.1 mg. Using a 100 ul syringe, immediately add two or more drops of assayed reference material to the flask and reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in micrograms per microliter from the net gain in weight. When compound purity is assayed at 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Transfer the stock standard solution into multiple TFE-sealed screw-cap bottles. Store with minimal headspace in the GC/MS VOA freezer. Once one of the bottles containing the standard solution has been opened, it may be used for at most one week.

Prepare fresh standards every two months for gases or for reactive compounds such as styrene. All other standards must be replaced after six months (four weeks for Method 524.2), or sooner if comparison with check standards indicates a problem.

Commercially prepared standards may be used at any concentration if they are certified by the manufacturer.

#### 3.3.1.2 Secondary dilution standards

Using stock standard solutions, prepare secondary dilution standards with methanol that contain the compounds of interest, either singly or mixed together.

Secondary dilution standards should be prepared at a level which yield working initial calibration standard concentrations at 20, 50, 100, 150 and 200 ug/L (except 10, 20, 50, 100 and 200 ug/L for the 1990 CLP Statement of Work and 0.2, 0.5, 1.0, 5.0, 20.0 ug/L for Method 524.2). Other initial calibration ranges may be employed to conform to specific protocols or if other conditions necessitate. A maximum volume of 10 ul of the secondary dilution standards should be used in the preparation of calibration standards, laboratory fortified blanks and laboratory fortified sample matrices to minimize the amount of methanol purged.

Secondary dilution standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

#### 3.3.1.3 Working calibration standards

Initial and continuing calibration standards are prepared in the syringe used to inject the standard into the purging device. The 50 ug/L (20 ug/L for Method 624 and 1.0 ug/L for Method 524.2) initial calibration standard is the continuing calibration

standard in most cases. Allow the standard solution to come to ambient temperature. Remove the plunger from a clean 5 mL (sometimes 25 mL for Method 524.2) syringe and close the valve. Pour reagent water into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the syringe valve and vent any residual air. Adjust the water volume to 5.0 mL (sometimes 25.0 mL for Method 524.2) minus the amount of standards to be added. Withdraw the plunger slightly, add 5.0 uL of the internal standard spiking solution and then add an appropriate volume of the secondary dilution standard and through the valve bore of the syringe. Close the valve and invert three times.

### 3.3.2 Internal Standards

The internal standard for Method 524.2 is fluorobenzene. Other methodologies use bromochloromethane, chlorobenzene- $d_3$  and 1,4-difluorobenzene as internal standards.

#### 3.3.2.1 Stock internal standard solutions

Commercially prepared internal standard solutions are used for stock solutions. The purities must be 99% or better. These are used to prepare internal standard spiking solutions, fortification solutions and for CLP medium level soil analyses.

#### 3.3.2.2 Internal standard spiking solutions

Using internal standard stock solutions, prepare spiking solutions with methanol so that the concentrations are 50 ug/uL (5.0 ug/uL for Method 524.2). Transfer the spiking standard solution into multiple TFE-sealed screw-cap vials. Store with no headspace in the GC/MS VOA refrigerator. Once one of the bottles has been opened, it may be used for at most one week. Prepare new spiking standards every 6 months (4 weeks for Method 524.2), or sooner if comparison of a freshly opened vial to the standard's recent history indicates a problem. These solutions are used to prepare calibration standards, laboratory fortified blanks, laboratory fortified sample matrices and for CLP medium level soil analyses.

### 3.3.3 Surrogate Standards (System Monitoring Compounds)

Surrogate standards for Method 524.2 are 1,2-dichlorobenzene- $d_4$  and 4-bromofluorobenzene (BFB). All other methodologies use 4-bromofluorobenzene, 1,2-dichloroethane- $d_4$  and toluene- $d_8$  as surrogate standards.

#### 3.3.3.1 Stock surrogate standard solutions

Commercially prepared surrogate standard solutions are used for stock solutions. The purities must be 99% or better. These are used to prepare surrogate standard calibration solutions and fortification solutions.

#### 3.3.3.2 Surrogate standard calibration solutions

Surrogate standard calibration solutions are prepared as target calibration solutions as in paragraphs 3.3.1.2 and 3.3.1.3.

#### 3.3.3.3 Surrogate standard spiking solutions

Surrogate standard spiking solutions are used in medium level soil analyses and are prepared, using the stock surrogate standard solutions, as in paragraph 3.3.2.2.

#### 3.3.4 Fortification Solutions

Fortification solutions combine all internal and surrogate standards into one spiking solution.

Using internal and surrogate standard stock solutions, prepare a fortification solution with methanol so that the concentrations are 50 ug/uL (5.0 ug/uL for Method 524.2). Transfer the fortification solution into multiple TFE-sealed screw-cap vials. Store with no headspace in the GC/MS VOA refrigerator. Once one of the bottles has been opened, it may be used for at most one week. Prepare a new fortification solution every six months (four weeks for Method 524.2), or sooner if comparison of a freshly opened vial to the history of the standards indicates a problem. These solutions are required to prepare reagent blanks, tuning check solutions and to fortify each sample.

#### 3.3.5 Tuning Check Compound

p-Bromofluorobenzene (BFB) is the VOA instrument performance check compound. The tuning check solution can be prepared from either the fortification solution or the surrogate standard spiking solution.

##### 3.3.5.1 Tuning check solution

Allow the solution to come to ambient temperature. Remove the plunger from a clean 5 mL syringe and close the valve. Pour reagent water into the syringe barrel to overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the syringe valve and vent any air. Adjust the water volume to 5.0 mL. Withdraw the plunger slightly and add 1.0 uL (0.5 uL for Method 524.2) of either solution through the valve bore of the syringe. Close the valve and invert three times.

### 3.3.6 Matrix Spike Compounds

The matrix spiking compounds for all methodologies (except 524.2, which uses all target compounds) are 1,1-dichloroethene, trichloroethene, benzene, toluene and chlorobenzene.

#### 3.3.6.1 Matrix spike stock solutions

Commercially prepared materials are used for matrix spike stock solutions. The purities must be 99% or better. These are used to prepare matrix spiking solutions.

#### 3.3.6.2 Matrix spiking solutions

Matrix spiking solutions are prepared, using the stock matrix spike solutions, as in paragraph 3.3.2.2. They are used to prepare sample matrix spikes and matrix spike duplicates.

## 3.4 Sample Preparation

### 3.4.1 Water Samples

The results of purgeable organics screening procedures determine what dilution (if any) is used for the initial analysis of water samples. All samples are allowed to reach ambient temperature before sampling through the septum or opening for dilution or analysis.

#### 3.4.1.1 Undiluted samples

Remove the plunger from a clean 5 mL (sometimes 25 mL for Method 524.2) syringe and close the valve. Rinse the syringe once or twice and then carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the valve and vent any residual air while adjusting the sample volume to 5.0 mL (sometimes 25.0 mL for Method 524.2).

This process of taking an aliquot destroys the validity of the sample for future analysis so, if there is only one VOA vial, the analyst should fill a second syringe at this time to protect against the possible loss of sample integrity. This second sample is maintained only until such time as the analyst has determined that the first sample has been analyzed properly and satisfies all quality assurance parameters. If an analysis is needed from the second syringe, it must be performed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

Withdraw the plunger of the syringe slightly and add 5.0 uL of the fortification solution through the valve bore of the syringe.

The addition of 5 uL of the fortification solution to 5 mL of sample is equivalent to a concentration of 50 ug/L (5 ug/L in Method 524.2) of each internal and surrogate standard. The addition of 5 uL of the fortification solution to 25 mL of sample in Method 524.2 is equivalent to a concentration of 1 ug/L of the internal and each surrogate standard.

#### 3.4.1.2 Diluted samples

The purgeable organics screening procedure shows the approximate concentrations of major sample components and any necessary dilution.

##### 3.4.1.2.1 CLP sample protocol

If a dilution of the sample was indicated, this dilution shall be made just prior to GC/MS volatile analysis of the sample. All steps in the dilution procedure must be performed without any delays until reaching the point at which the diluted sample is in a gas-tight syringe with the syringe valve closed.

All dilutions are made in class A volumetric flasks (10 mL to 100 mL). Select the flask that allows for the necessary dilution. Intermediate dilutions may be necessary for extremely large dilutions.

Calculate the approximate volume of reagent water which will be added to the volumetric flask selected and add slightly less than this quantity of reagent water to the flask.

Inject the proper aliquot from the syringe prepared in paragraph 3.4.1.1 (without the fortification solution) into the volumetric flask. Aliquots of less than 1 mL increments are prohibited. Dilute the flask to the volumetric mark with reagent water. Cap the flask, invert, and shake three times.

Fill a 5 mL syringe with the diluted sample and prepare it as in paragraph 3.4.1.1. If this is an intermediate dilution, use it (without the fortification solution) and repeat the procedure as necessary to achieve larger dilutions.

##### 3.4.1.2.2 Non-CLP sample protocols

Remove the plunger from a clean 5 mL syringe and close the valve. Pour reagent water into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open the syringe valve and vent any residual air. Adjust the water volume to

5.0 mL minus the amount of the sample aliquot to be added. Take the sample aliquot by piercing the closed vial septum cap with a sampling microsyringe. Withdraw the plunger slightly and add the appropriate aliquot of the sample (determined by the screening procedure) and 5.0 uL of the internal standard spiking solution through the valve bore of the syringe. Close the valve and invert three times.

### 3.4.2 Soil Samples

The results of volatile screening procedures determine whether the low or medium level method must be followed and what dilution (if any) is used for the initial analysis. Soil, sludge, solid and semi-solid samples are henceforth referred to as soil samples.

#### 3.4.2.1 Low level soil method

The low level soil method is based on a soil sample mixed with reagent water containing the fortification solution. This method is used if screening procedures indicate that the most abundant component is less than five times the highest calibration standard concentration level.

The sample for volatile organics consists of the **entire** contents of the sample container. Do not discard any supernatant liquids. Thoroughly mix the contents of the sample container with a narrow metal spatula. Weigh five grams of the homogeneous sample (or to a minimum of one gram if screening procedures suggest) into a clean purge chamber, using a top-loading balance. Note and record (in the GC/MS VOA Logbook for that system) the actual weight to the nearest 0.1 g. **Immediately** install the chamber onto the purge and trap device.

Without delay, tare a labelled aluminum dish, then weigh 5-10 g of the homogeneous sample (from the same container used to take the sample) into the dish and record both weights in the GC/MS %Moisture Logbook. Dry overnight at 105° C and allow to cool in a dessicator before weighing. Record the dry weight and calculated percent moisture in the GC/MS %Moisture Logbook. Also record the percent moisture in the GC/MS VOA Logbook for that system.

To prepare the reagent water containing the fortification solution, remove the plunger from a clean 5 mL syringe and close the valve. Pour reagent water into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the water. Invert the syringe, open valve and vent any residual

air. Adjust the water volume to 5.0 mL. Withdraw the plunger slightly and add 5.0 uL of the fortification solution through the valve bore of the syringe. Close the valve and invert three times.

#### 3.4.2.2 Medium level soil methods

The medium level soil methods are based on extracting the soil sample with methanol and adding an aliquot of the methanol extract to reagent water containing the fortification solution. These methods are used if screening procedures indicate that the most abundant component is greater than five times the highest calibration standard concentration level.

##### 3.4.2.2.1 CLP sample protocol

The sample for volatile organics consists of the **entire** contents of the sample container. Do not discard any supernatant liquids. Mix the contents of the sample container with a narrow metal spatula. Weigh 4 g (wet weight) into a tared 40 mL VOA vial. Use a top-loading balance. Note and record (in the GC/MS VOA Logbook for that system) the actual weight to the nearest 0.1 g. Determine the percent moisture as in paragraph 3.4.2.1.

Quickly add 9.5 mL of methanol to the vial. Then add 0.5 mL of the surrogate standard spiking solution. Cap and shake for 2 minutes. NOTE: The steps above must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

Using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be discarded. Transfer approximately 1 mL of the reagent methanol to a GC vial for use in the method blank for each day on which medium level soil sample extractions are performed. These extracts may be stored in the GC/MS VOA refrigerator prior to analysis.

Use the estimated concentration range of the sample from the screening procedure to determine the appropriate volume. All dilution must keep the response of the major constituents in the upper half of linear range of the curve.

Remove the plunger from a 5 mL Luerlock gas-tight syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.95 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of sample and standards. Add 5.0 uL of the

internal standard spiking solution. Also add the volume of methanol extract determined in the previous paragraph and a volume of clean methanol to total 50 uL (excluding methanol in standard spiking solution).

#### 3.4.2.2.2 Non-CLP sample protocol

The sample for volatile organics consists of the **entire** contents of the sample container. Do not discard any supernatant liquids. Thoroughly mix the contents of the sample container with a narrow metal spatula. Weigh 5 g into a tared 40 mL VOA vial. Use a top-loading balance. Note and record (in the GC/MS VOA Logbook for that system) the actual weight to the nearest 0.1 g. Quickly add 5.0 mL of purge-and-trap methanol to the vial. Cap and shake for 2 minutes and let the solids settle. Determine the percent moisture as in paragraph 3.4.2.1. NOTE: The steps above must be performed rapidly to avoid loss of volatile organics. These steps must be performed in a laboratory free of solvent fumes.

Using a disposable pipette, transfer approximately 1 mL of extract into a GC vial for storage. The remainder may be discarded. Transfer approximately 1 mL of the reagent methanol to a GC vial for use in the method blank for each day on which medium level soil sample extractions are performed. These extracts may be stored in the GC/MS VOA refrigerator prior to analysis.

Use the estimated concentration range of the sample from the screening procedure to determine the appropriate volume. All dilution must keep the response of the major constituents in the upper half of linear range of the curve.

Remove the plunger from a 5 mL Luerlock gas-tight syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to 4.95 mL. Pull the plunger back to 5.0 mL to allow volume for the addition of sample and standards. Add 5.0 uL of the fortification solution. Also add the volume of methanol extract determined in the previous paragraph and a volume of clean methanol to total 50 uL (excluding methanol in fortification solution).

#### 3.4.3 Sample Matrix Spikes

Matrix spikes, matrix spike duplicates and laboratory fortified sample matrices (Method 524.2 only) are samples of any type to

which target analytes are added to assess whether the sample matrices contribute bias to the analytical results.

#### 3.4.3.1 CLP medium-level soil sample matrix spike

Follow the sample preparations described in paragraph 3.4.2.2.1 except add 9.0 mL of methanol to the vial, then add 0.5 mL each of the surrogate standard spiking solution and the matrix spiking solution to the vial.

#### 3.4.3.2 Non-CLP and CLP water or low-level soil matrix spikes.

Follow the sample preparations described in 3.4.1.1, 3.4.1.2, 3.4.2.1 and 3.4.2.2. In addition, add 5.0 uL of the matrix spiking solution to the syringe (in Method 524.2, add an appropriate amount of the secondary dilution calibration standard to create a laboratory fortified sample matrix of 5 ug/L, 1 ug/L for 25 mL samples).

### 3.5 Blank Preparation

#### 3.5.1 Laboratory Blank

An aliquot of reagent water or blank soil is treated exactly as a sample including exposure to all glassware, equipment, reagents, internal standards and surrogate standards. It is used to determine if method analytes or other interferences are present in the laboratory environment, reagents or apparatus.

#### 3.5.2 Field Blank

Reagent water or blank soil is placed in a sample container at the sample site and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, transportation to the laboratory and all analytical procedures. It is used to determine if method analytes or other interferences are present in the field environment.

#### 3.5.3 Trip Blank

Reagent water or blank soil is placed in a sample container in the laboratory, sent with containers for sampling (never opened) and treated as a sample in all respects, including exposure to transportation to and from the sample site, sampling site conditions, storage, preservation and all analytical procedures.

#### 3.5.4 Holding Blank

For CLP sample lots, a holding blank is prepared from reagent water or blank soil and carried through the holding period and

analysis protocol as a sample. It serves as a check against contamination by diffusion of volatile organics through the septum seal into the sample during storage and handling.

### 3.5.5 Laboratory Fortified Blank

Known quantities of target analytes are added to reagent water in the laboratory. It is analyzed exactly like a sample in Method 524.2.

## 4.0 INSTRUMENT OPERATING CONDITIONS

### 4.1 Purge and Trap Device

#### 4.1.1 Purge Conditions

Purge gas: helium  
Purge time: 11.0 +/-0.1 minutes  
Purge flow rate: 36 mL/min (25-40 mL/min allowed)  
Sampler purge temperature: ambient, 40° C, or 85° C  
Trap purge temperature: less than 22° C  
Sampler transfer temperature: 80° C

#### 4.1.2 Desorb Conditions

Pre-heat temperature: 160° C  
Desorb temperature: 180° C  
Desorb time: 4.0 +/-0.1 minutes  
Desorb transfer temperature: 80° C

#### 4.1.3 Trap Reconditioning Conditions

Reconditioning temperature: 200° C  
Reconditioning time: 7.0-10.0 minutes

### 4.2 Gas Chromatograph

Recommended gas chromatographic analytical conditions:

Carrier gas: helium  
Flow rate: 2.6 mL/min  
Injection port temperature: 200° C  
Jet Separator temperature: 210° C (if installed)  
MS Interface temperatures: 220° C  
Initial temperature: -10° C

Initial ramp rate: 20° C/min  
Secondary temperature: 30° C  
Secondary ramp rate: 8° C/min  
Final temperature: 200° C

#### 4.3 Mass Spectrometer

Required mass spectrometric analytical conditions:

Electron energy: 70 volts (nominal)  
Mass range: 35-300 amu  
Scan time: To give at least 5 scans per peak, not to exceed 1 second per scan

### 5.0 CALIBRATION

#### 5.1 Instrument Performance Calibration

Prior to the analysis of any samples, blanks or calibration standards, it must be established that the GC/MS system meets the mass spectral ion abundance criteria for the tuning check solution containing p-bromofluorobenzene (BFB).

##### 5.1.1 Manufacturer's Internal Calibration Compound

Neat PFTBA (perfluorotributylamine) is used to monitor the MS source parameters when tuning. It is introduced through a valved induction tube, by vaporization directly into the source chamber.

##### 5.1.2 System Performance Calibration Compound

BFB (p-bromofluorobenzene) is introduced by analyzing the tuning check solution (3.3.5.1) as a sample, except for GC temperature programming alterations which are allowed to expedite the procedure. This exception is not allowed in the 1990 CLP SOW.

##### 5.1.3 Ion abundance criteria

There are three different criteria for BFB tuning. Traditional criteria are used for Method 624, Method 8260 and the 1988 (with 1989 additions) CLP protocol. New criteria are in effect for the CLP SOW issued in 1990. Method 524.2 has yet different criteria. Volatile procedures in this laboratory follow the traditional criteria except when required or requested otherwise.

#### 5.1.3.1 Traditional tuning criteria

A mass spectrum from the analysis of the tuning solution must meet the ion abundance criteria given below.

#### BFB KEY IONS AND ION ABUNDANCE CRITERIA

##### Mass Ion Abundance Criteria

---

50 15.0 - 40.0 percent of mass 95  
75 30.0 - 60.0 percent of mass 95  
95 base peak, 100 percent relative abundance  
96 5.0 - 9.0 percent of mass 95  
173 less than 2.0 percent of mass 174  
174 greater than 50 percent of mass 95  
175 5.0 - 9.0 percent of mass 174  
176 95.0 - 101.0 percent of mass 174  
177 5.0 - 9.0 percent of mass 176

#### 5.1.3.2 Method 524.2 tuning criteria

A mass spectrum from the analysis of the tuning solution must meet the ion abundance criteria given below. An average spectrum across the GC peak may be used to evaluate the performance of the system.

#### BFB KEY IONS AND ION ABUNDANCE CRITERIA

##### Mass Ion Abundance Criteria

---

50 15 to 40% of mass 95  
75 30 to 80% of mass 95  
95 Base Peak, 100% Relative Abundance  
96 5 to 9% of mass 95  
173 < 2.0 percent of mass 174  
174 > 50% of mass 95  
175 5 to 9% of mass 174  
176 > 95 but < 101% of mass 174  
177 5 to 9% of mass 176

#### 5.1.3.3 New CLP (1990) tuning criteria

The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB. All instrument conditions must be identical to those used in sample analysis. A mass spectrum from the analysis of the tuning solution must meet the ion abundance criteria given below.

## BFB KEY IONS AND ION ABUNDANCE CRITERIA

### Mass Ion Abundance Criteria

---

50 8.0 - 40.0 percent of mass 95  
75 30.0 - 66.0 percent of mass 95  
95 base peak, 100 percent relative abundance  
96 5.0 - 9.0 percent of mass 95  
173 less than 2.0 percent of mass 174  
174 50.0 - 120.0 percent of mass 95 (see note)  
175 4.0 - 9.0 percent of mass 174  
176 93.0 - 101.0 percent of mass 174 (see note)  
177 5.0 - 9.0 percent of mass 176

Note: All ion abundances must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 or m/z 176 may be up to 120 or 121.2 percent that of m/z 95.

### 5.2 Initial Calibration

Prior to the analysis of samples and blanks, and after the tuning criteria have been met, each GC/MS system must be calibrated to determine instrument sensitivity and the linearity of response.

#### 5.2.1 Calibration Standard Analyses

Analyze at least five concentrations of target analytes following procedures paragraphs 3.3.1.3, 6.3 and section 4.0. Process the initial calibration (name sequentially in format shown in overall GC/MS procedure) through CBUPD and CBRPT which automatically calculates and reports the relative response factors, average response factors and the relative standard deviations using the formulas outlined in the following paragraphs.

#### 5.2.2 Relative Response Factor Calculation

Tabulate the area response of the characteristic ions in the extracted ion current profile against concentration for each compound and internal standard and calculate relative response factors (RRF) for each compound using the following equation:

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

- $A_x$  = Area of the characteristic ion for the target compound  
 $A_{is}$  = Area of the characteristic ion for the specific internal standard  
 $C_{is}$  = Concentration of the internal standard  
 $C_x$  = Concentration of the compound to be measured

### 5.2.3 Average RRF and Relative Standard Deviation Calculation

The average relative response factor ( $\overline{RRF}$ ) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve.

$$\%RSD = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

### 5.2.4 Initial Calibration Response Factor Criteria

Response factor criteria are used for volatile target compounds and surrogate standards. The variant criteria which exist for Methods 624, 8260, 524.2 and the CLP Statements of Work issued in 1988 and 1990 are listed in the paragraphs below. The 1988 CLP criteria are in effect unless otherwise specified.

#### 5.2.4.1 CLP response factor criteria

The compounds with entries listed below must meet the minimum RRF and maximum %RSD criteria for the initial calibration.

Volatile Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
Chloromethane	.300	.010		
Bromomethane		.100		20.5
Vinyl Chloride		.100	30.0	20.5
Chloroethane		.010		
Methylene Chloride		.010		
Acetone		.010		
Carbon Disulfide		.010		
1,1-Dichloroethene		.100	30.0	20.5
1,1-Dichloroethane	.300	.200		20.5
1,2-Dichloroethene (total)		.010		
Chloroform		.200	30.0	20.5
1,2-Dichloroethane		.100		20.5
2-Butanone		.010		

Volatile Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
1,1,1-Trichloroethane		.100		20.5
Carbon Tetrachloride		.100		20.5
Bromodichloromethane		.200		20.5
1,2-Dichloropropane		.010	30.0	
cis-1,3-Dichloropropene		.200		20.5
Trichloroethene		.300		20.5
Dibromochloromethane		.100		20.5
1,1,2-Trichloroethane		.100		20.5
Benzene		.500		20.5
trans-1,3-Dichloropropene		.100		20.5
Bromoform	.250	.100		20.5
4-Methyl-2-Pentanone		.010		
2-Hexanone		.010		
Tetrachloroethene		.200		20.5
1,1,2,2-Tetrachloroethane	.300	.500		20.5
Toluene		.400	30.0	20.5
Chlorobenzene	.300	.500		20.5
Ethylbenzene		.100	30.0	20.5
Styrene		.300		20.5
Xylene (total)		.300		20.5
1,2-Dichlorobenzene-d <sub>4</sub>		.010		
Bromofluorobenzene		.200		20.5
Toluene-d <sub>8</sub>		.010		

Note that the 1990 SOW allows two compounds with a RRF>.010 requirement and a specified %RSD to be noncompliant. However the RRF's for those two compounds must be > or =.010, and the %RSD must be < or =40.0 for the initial calibration to be acceptable.

#### 5.2.4.2 Method 624 response factor criteria

The RSD of all analytes and surrogates must be less than or equal to 35%.

#### 5.2.4.3 Method 8260 response factor criteria

The criteria are identical to the 1988 CLP criteria.

#### 5.2.4.4 Method 524.2 response factor criteria

The Relative Standard Deviation of all analytes and surrogates must be less than or equal to 20%. As an alternative to calculating mean response factors and applying the percent RSD criteria, the Aquarius software can be utilized (through CBUPD and CBCAL) to generate a second or third order regression calibration curve.

### 5.3 Continuing Calibration

Prior to the analysis of samples and the required blanks, and after the daily tuning criteria have been met, a check of the calibration curve (referenced to the initial calibration) must be performed once every working shift on each GC/MS system, unless an initial calibration was performed.

#### 5.3.1 Calibration Standard Analysis

Analyze a continuing check standard of target analytes prepared as in paragraph 3.3.1.3, using the instrument conditions described in section 4.0 and the procedures outlined in paragraph 6.3. Collect the acquisition data and store in the data system. Process the continuing calibration through CBCHK (paragraph 3.4.7 in the GC/MS overview) which automatically calculates the continuing response factors, and the percent difference between them and the average response factors generated by the initial calibration.

#### 5.3.2 Continuing Calibration Response Factor Criteria

The continuing calibration criteria are described in the following paragraphs. Check the relative response factors of those compound for which minimum RRF values have been established. If these criteria are met, the relative response factors for all compounds are calculated and reported. Compare the percent difference with any criteria listed in the paragraphs below. The 1988 CLP criteria are in effect unless otherwise specified.

##### 5.3.2.1 Method 624 calibration curve verification criteria

The calibration curve verification is performed by analysis of a 20 ug/L QC check sample. For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in the table below. Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve or RF must be prepared for that parameter.

Parameter	Range for Q (ug/L)
Benzene	12.8-27.2
Bromodichloromethane	13.1-26.9
Bromoform	14.2-25.8
Bromomethane	2.8-37.2
Carbon tetrachloride	14.6-25.4
Chlorobenzene	13.2-26.8
Chloroethane	7.6-32.4
2-Chloroethylvinyl ether	D-44.8
Chloroform	13.5-26.5
Chloromethane	D-40.8
Dibromochloromethane	13.5-26.5
1,1-Dichloroethane	14.5-25.5
1,2-Dichloroethane	13.6-26.4
1,1-Dichloroethene	10.1-29.9
trans-1,2-Dichloroethene	13.9-26.1
1,2-Dichloropropane	6.8-33.2
cis-1,3-Dichloropropene	4.8-35.2
trans-1,3-Dichloropropene	10.0-30.0
Ethyl benzene	11.8-28.2
Methylene chloride	12.1-27.9
1,1,2,2-Tetrachloroethane	12.1-27.9
Tetrachloroethene	14.7-25.3
Toluene	14.9-25.1
1,1,1-Trichloroethane	15.0-25.0
1,1,2-Trichloroethane	14.2-25.8
Trichloroethene	13.3-26.7
Trichlorofluoromethane	9.6-30.4
Vinyl chloride	0.8-39.2

Q = Concentration measured in check standard, in ug/L  
D = Detected; result must be greater than zero

5.3.2.2 CLP calibration check criteria

The compounds with entries listed below must meet the minimum RRF and maximum %Difference criteria for the continuing calibration.

Volatile Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
Chloromethane	.300	.010		
Bromomethane		.100		25.0
Vinyl Chloride		.100	25.0	25.0
Chloroethane		.010		
Methylene Chloride		.010		
Acetone		.010		
Carbon Disulfide		.010		
1,1-Dichloroethene		.100	25.0	25.0
1,1-Dichloroethane	.300	.200		25.0
1,2-Dichloroethene (total)		.010		
Chloroform		.200	25.0	25.0
1,2-Dichloroethane		.100		25.0
2-Butanone		.010		
1,1,1-Trichloroethane		.100		25.0
Carbon Tetrachloride		.100		25.0
Bromodichloromethane		.200		25.0
1,2-Dichloropropane		.010	25.0	
cis-1,3-Dichloropropene		.200		25.0
Trichloroethene		.300		25.0
Dibromochloromethane		.100		25.0
1,1,2-Trichloroethane		.100		25.0
Benzene		.500		25.0
trans-1,3-Dichloropropene		.100		25.0
Bromoform	.250	.100		25.0
4-Methyl-2-Pentanone		.010		
2-Hexanone		.010		
Tetrachloroethene		.200		25.0
1,1,2,2-Tetrachloroethane	.300	.500		25.0
Toluene		.400	25.0	25.0
Chlorobenzene	.300	.500		25.0
Ethylbenzene		.100	25.0	25.0
Styrene		.300		25.0
Xylene (total)		.300		25.0
1,2-Dichlorobenzene-d <sub>4</sub>		.010		
Bromofluorobenzene		.200		25.0
Toluene-d <sub>8</sub>		.010		

Note that the 1990 SOW allows two compounds with a RRF>.010 requirement and a specified %Diff to be noncompliant, however, the RRF's must be > or =.010, and the %Diff must be < or =40.0 for the continuing calibration to be acceptable.

5.3.2.3 Method 8260 continuing response factor criteria  
The criteria for Method 8260 are identical to the 1988 CLP criteria.

5.3.2.4 Method 524.2 continuing response factor criteria  
The response factor for each analyte and surrogate standard must be within 30% of the mean value measured in the initial calibration. If a second or third order regression is used, the point from the check standard for each analyte and surrogate must fall, within the analyst's judgement, on the initial calibration curve.

## 6.0 ANALYSIS

### 6.1 Analytical Shift

The various methodologies require that all quality assurance operations and sample analyses be performed within certain time restrictions to maintain a substantive association to calibration and quality assurance operations.

#### 6.1.1 Time Definition

The start of the analytical shift occurs at the injection time of the BFB tuning check solution which **passes tuning criteria**. All samples and quality assurance operations must be injected before the shift duration (from the injection of the tuning solution) elapses.

#### 6.1.2 Methodology Specifications

The durations of analytical shifts vary between methodologies. Method 624 designates a shift as a "day", which is a continuous operational run no longer than a 24 hour period. Twelve hours is specified as the analytical shift for Method 8260 and CLP methodologies, whereas Method 524.2 uses eight hours as the shift duration.

#### 6.1.3 Shift Quality Assurance Requirements

Before samples are analyzed in a shift, the following quality assurance criteria must be passed: a 4-bromofluorobenzene tune, an initial or continuing calibration and a laboratory blank analysis. These analyses must be performed in that particular order. For Method 524.2, a fortified laboratory blank must be analyzed each shift.

## 6.2 Target Analytes

Individual volatile target analytes are listed below with the practical quantitation limits (PQL's), in ug/L or ug/Kg, achievable on Kiber Analytical instruments at all times. We routinely achieve significantly lower limits. The PQL's listed are based upon full-weight, undiluted samples. Diluted and reduced-weight samples result in quantitation limits which are higher by the factor of dilution and/or weight reduction.

Detection limit studies are performed whenever the analytical system is grossly modified or every six months, whichever is sooner. Normal reportable detection limits have been determined by the procedure published in 40 CFR 136, Appendix B. Detection limits are also determined by the Hubaux and Vos procedure (*Anal. Chem.*, 42, 849 (1970) and Kiber Analytical's positive definite procedure (which is performed using all ions greater than 10% base peak abundance).

Limits for Method 524.2 analyses are noted with a "D". Use of 25mL spargers lowers these limits by a factor of five. Analytes that require heated purging are designated with an "-H". An asterisk (\*) denotes a compound which hydrolyzes in water. All sample results for this compound should be denoted as estimated.

Analytes which are on a target list are noted with the following codes: P=Priority Pollutant, T=Toxic/Target Compound, X=Appendix IX, D=drinking water, C=California list, R=LDRL, and L=TCLP.

Please note that many non-target volatiles can be analyzed if an acceptable standard is obtained and it can be shown that Kiber Analytical configurations allow accurate, reproducible results.

VOLATILE ANALYTE	PQL	LIST CODES
Acetone	50	TX R
Acetonitrile-H	50	X
Acrolein-H	100	P X
Acrylamide-H	200	
Acrylonitrile-H	25	P X
Allyl chloride (3-Chloro-1-propene)	10	X C
Benzene	5,1D	PTXD L
Bromobenzene	1D	D
Bromochloromethane	2D	D
Bromodichloromethane (Dichlorobromomethane)	5,1D	PTXDC
Bromoform	5,1D	PTXDC
Bromomethane (Methyl bromide)	5,1D	PTXDC

VOLATILE ANALYTE	PQL	LIST CODES
2-Butanone (Methyl ethyl ketone), (MEK)	50	TX RL
n-Butyl alcohol (n-Butanol)-H	200	R
n-Butylbenzene	2D	D
sec-Butylbenzene	5D	D
tert-Butylbenzene	2D	D
Carbon disulfide	5	TX R
Carbon tetrachloride	5, 2D	PTXDCRL
Chlorobenzene	5, 1D	PTXDCRL
Chloroethane	10, 1D	PTXDC
2-Chloroethyl vinyl ether*	100	P C
Chloroform	5, 2D	PTXDC L
Chloromethane (Methyl chloride)	10, 1D	PTXDC
Chloroprene (2-Chloro-1,3-butadiene)	100	X C
2-Chlorotoluene	1D	D
4-Chlorotoluene	1D	D
Dibromochloromethane (Chlorodibromomethane)	5, 1D	PTXDC
1,2-Dibromo-3-chloropropane (DBCP)	20, 2D	XDC
1,2-Dibromoethane (Ethylene dibromide), (EDB)	5, 1D	XDC
Dibromomethane (Methylene bromide)	5, 2D	XDC
1,2-Dichlorobenzene (o-Dichlorobenzene)	5D	D
1,3-Dichlorobenzene (m-Dichlorobenzene)	5D	D
1,4-Dichlorobenzene (p-Dichlorobenzene)	5D	D
trans-1,4-Dichloro-2-butene	10	X C
Dichlorodifluoromethane	200, 20D	XDC
1,1-Dichloroethane	5, 1D	PTXDC
1,2-Dichloroethane	5, 1D	PTXDC L
1,1-Dichloroethene (1,1-Dichloroethylene)	5, 2D	PTXDC L
cis-1,2-Dichloroethene (c1,2-Dichloroethylene)	2D	D
trans-1,2-Dichloroethene (t1,2-Dichloroethylene)	5, 2D	P XDC
1,2-Dichloroethene (total)	5	T
1,2-Dichloropropane	5, 1D	PTXDC
1,3-Dichloropropane	1D	D
2,2-Dichloropropane	2D	D
1,1-Dichloropropene (1,1-Dichloropropylene)	2D	D
cis-1,3-Dichloropropene (c1,3-Dichloropropylene)	5, 1D	PTXDC
trans-1,3-Dichloropropene (t1,3-Dichloropropylene)	5, 1D	PTXDC
Diethyl ether (Ethyl ether)	10	R
1,4-Dioxane-H	200	X
Ethylbenzene	5, 1D	PTXD R
Ethyl methacrylate	5	X

VOLATILE ANALYTE	PQL	LIST CODES
Hexachlorobutadiene	20D	D
n-Hexane	5	
Ethanol (Ethyl alcohol)	1000	
Ethyl acetate	200	R
2-Hexanone	20	TX
Isobutyl alcohol-H	50	X R
Isopropylbenzene (Cumene)	1D	D
4-Isopropyltoluene	2D	D
Methacrylonitrile	20	X
Methyl tert-butyl ether (MTBE)	1D	D
Methylene chloride (Dichloromethane)	5,2D	PTXDCR
Methyl iodide (Iodomethane)	5	X C
Methyl methacrylate	50	X
4-Methyl-2-pentanone (Methyl isobutyl ketone) (MIBK)	20	TX R
Naphthalene	2D	D
Paraldehyde	500	
Pentachloroethane	20	X C
Propionitrile-H	50	X
n-Propylbenzene	1D	D
Pyridine-H	500	X RL
Styrene	5,1D	TXD
1,1,1,2-Tetrachloroethane	5,1D	XDC
1,1,2,2-Tetrachloroethane	5,1D	PTXDC
Tetrachloroethene (Perchloroethylene)	5,2D	PTXDCRL
Toluene (Methylbenzene)	5,1D	PTXD R
1,2,3-Trichlorobenzene	5D	D
1,2,4-Trichlorobenzene	5D	D
1,1,1-Trichloroethane	5,1D	PTXDCR
1,1,2-Trichloroethane	5,1D	PTXDC
Trichloroethene (Trichloroethylene)	5,1D	PTXDCRL
Trichlorofluoromethane (Freon 11)	5,1D	XDCR
1,2,3-Trichloropropane	10,1D	XDC
1,1,2-Trichlorotrifluoroethane (Freon 113)	5	R
1,2,4-Trimethylbenzene (Pseudocumene)	1D	D
1,3,5-Trimethylbenzene (Mesitylene)	1D	D
Vinyl acetate (CLP target before 1990 only)	5	TX
Vinyl chloride	10,2D	PTXDC L
m- & p-Xylene (1,3- & 1,4-Dimethylbenzene)	1D	D
o-Xylene (1,2-Dimethylbenzene)	1D	D
Xylenes (total)	5	TX R

## 6.3 Sample Analysis Operations

### 6.3.1 Documentation

All instrumental and data system parameters unique to the analytical runs must be documented in the GC/MS Logbook for that particular instrument. The logs are bound and contain information on all runs, successful or not.

In addition, all quality assurance results are generated after analysis and placed in a GC/MS Quality Assurance Notebook which is maintained for each instrument.

### 6.3.2 Data System Operations

#### 6.3.2.1 Batch analysis arrangement

Groups of analytical runs are set up in a sequence file using the BEDIT program. Sequence file namrs use the format SEQ\_XX and can be reused. Run files namrs are named with five-digits, consecutively ordered, from 20000 on GC/MS Instrument #1 and from 40000 on GC/MS Instrument #2, prescribed with ">". Follow procedures outlined in paragraphs 3.2.1 and 3.2.2 of the overall GC/MS protocol to edit BEDIT and generate a BLIST output.

#### 6.3.2.2 Batch analysis

Sequence files containing batch runs are controlled by the BAMON program (paragraph 3.2.3 of GC/MS overview). Care should be taken to **start** a sequence close to the time that a purging cycle ends, so as to conserve cryogenic liquid and prevent excessive exposure of the column fixtures to subambient temperatures.

It is desirable to pause the sequence file through BAMON after each run involving tuning, calibration and laboratory blank analyses. This pause allows a time interval for checking the resultant data against the required quality assurance criteria and averts useless analyses in those cases where the criteria have not been met.

### 6.3.3 Sparger Preparation

#### 6.3.3.1 Solution introduction

Load the solution onto the automatic purging module by opening or removing the syringe valve and discharging the contents of the syringe through the sparge tube valve into a empty clean sparger (aqueous) or a clean sparger containing a measured soil sample. Immediately close the sparge tube valve. Multiple solutions can be loaded but must be purged and desorbed within 12 hours.

#### 6.3.3.2 Sample heating

Analyses of low-level soil samples or analytes which require heat (see paragraph 6.2) utilize the sample heating unit. Place the heating jacket around the sparge tube and fasten the closure. Set the unit temperature to 40° C for low-level soil analyses or to 85° C to analyze any matrix in which analytes requiring heat are targets.

### 6.3.4 Analytical Operations

#### 6.3.4.1 Integrated instrument operations

The automatic sampler, purge and trap, gas chromatograph, mass spectrometer and data system are configured so that sample purging, sample desorption, instrumental operations and data system acquisition can commence only when all components are at their proper setpoints and conditions.

#### 6.3.4.2 Purge and trap unit operation

Make sure the unit temperatures are at the setpoints listed in paragraph 4.1. Automatic and purging of consecutive spargers can be performed if the AUTO Key is set to ON and the AUTO SPL is set to a higher number than the AUTO SPL VIEW indicates. Manually start the purge of each run involving tuning, calibration, laboratory blank, and the initial sample analysis. This allows time for checking the previous analysis against the quality assurance criteria and averts useless analyses if the criteria have not been met. Press the RUN key to start purging. The unit holds after the purge cycle and does not enter the desorb mode until all system components are ready.

#### 6.3.4.3 GC/MS operation

Setpoints for the gas chromatograph and mass spectrometer are set within the METHOD file. This set file is included in the sequence file setup using program BEDIT. A hard copy of the method can be obtained by invoking the LMETH program and should be saved for inclusion with sample paperwork if individual GC/MS parameters are not given in the GC/MS Logbook. All GC/MS

instrumental operations are controlled by BAMON (paragraph 3.2.3 in the GC/MS Overview).

## 7.0 POST-ACQUISITION DATA PROCESSING

Post-acquisition processing is performed on all data files to validate the automatic data processing, check for proper chromatographic operation, generate quality assurance and compare QA results to required criteria, and finalize target analyte identifications and quantitations.

### 7.1 Data File Configuration

#### 7.1.1 Data File Designations

The five-digit acquisition file number is prescribed with particular characters to designate the type of file. The ">" symbol signifies the raw data file acquired during GC/MS analysis. A scan directory file is normally generated automatically at the end of acquisition, is designated by an initial "<" character, and must be present in order to fully process the analysis. The corresponding Quant Output file is ordinarily designated by the character "^" in front of the number. If distinct quantitations are performed, use an alternative descriptor "V" for the first additional namr and then any unique descriptor such as A or Q (but not S). During softkey CLP forms generation an "=" file is created which contains all deliverable contract information. An example of allied files from the same run would be: >42173, <42173 , ^42173, V42173 and =42173.

#### 7.1.2 File Corruption and Modification

Occasionally data file truncation or directory file corruption occurs. Data file abortion occurs when the run is terminated by power disruption, manual request or when the analysis runs out of disc space during acquisition. Directory file corruption occurs when the data file is terminated for the above reasons or the files are accessed at the same time that the directory file is being generated (immediately after acquisition). These events prevent any Quant Output files from being created at that time.

The analyst can regenerate a nonexistent or corrupted directory file from the raw data file by invoking program FIXDA. The data file can then be accessed to determine if the acquisition satisfies all analytical requirements (including run-length long enough to include the last target analyte), or whether it should

be rerun. Quant Output files should be created through program QT.

## 7.2 Generic Manual Quant Output Validation Procedure

The manual validation process is performed by total ion chromatogram (TIC) examination and processing the target analyte outputs through the QAREA and QDEL programs (paragraphs 3.3.5 and 3.3.7. All target "hits" are checked for confirmation of proper identification, retention time, proper integration and interferences.

### 7.2.1 Total Ion Chromatogram Examination

The total ion chromatogram (TIC) trace produced in the Medium Quant Output format should be appraised. In general, the chromatogram should appear normal with all predicted standard peaks present. Flattened or tailing peaks, poor peak resolution, an aberrant baseline, a lack (or dropoff) of signal, or a shortened scan time could indicate that the analytical system has malfunctioned and operations should be suspended until the cause of the failure is determined and corrected.

A GC column overburden is indicated by gross abundances in the total ion chromatogram. Indications of overloading are retarded retention times, flattened or rounded peak apices, poor peak resolution in regions of high response and "hump-o-grams". If column overloading occurs, the sample should be reanalyzed at a greater dilution.

The presence of relatively high molecular weight components is exhibited by a response or elevated baseline at the end of the temperature program. Note significant occurrences of this situation (and note the sparge port used for analysis) as they necessitate system cleaning before certain volatile analyses.

### 7.2.2 Target Analyte Identification Confirmation

Two criteria must be satisfied to verify identification: (1) elution of a sample component at the same relative retention time (RRT) as that of the standard component and (2) correspondence of the sample component and the standard component mass spectrum.

#### 7.2.2.1 Retention time requirements

The sample component RRT must compare within +/- 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment

of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

#### 7.2.2.2 Spectral requirements

For comparison of sample and standard component mass spectra, the reference spectra must have been previously obtained from Kiber Analytical's GC/MS instrumentation. The sample spectrum is accessed (by entering "P" as a QAREA command) and compared to the reference spectrum (by then entering "R"). The requirements for qualitative verification by comparison of mass spectra are as follows:

7.2.2.2.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum. In a special case for CLP, the 72 ion must be present in the spectrum of 2-Butanone for positive identification.

7.2.2.2.2 The relative intensities of ions specified in the above paragraph must agree within +/-20% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be within 30 and 70 percent.

7.2.2.2.3 Ions greater than 10% in the **sample** spectrum but not present in the **standard** spectrum must be considered and accounted for by the analyst making the comparison.

#### 7.2.2.3 Interpreter judgement

Even if a compound cannot be verified by all of the criteria above, an identification can be validated through the technical judgement of the mass spectral interpretation specialist.

#### 7.2.2.4 Deletion of unconfirmed analytes

The deletion of false positives generated by the Quant process is performed through the QDEL program. If false positives were found during QAREA operations, exit QAREA **without archiving** and delete any unconfirmed "hits" by using QDEL. The quant file should then be archived through ARCV.

#### 7.2.3 Quantitation Verification

The automated integration of each reported "hit" should be viewed through QAREA and when not accurate, should be manually corrected following the guidelines in the description of the QAREA program.

The analyst should note any interferences with the quantitation ion and, if possible, remove the contributing interferent without distorting the target integration. If this is not possible, the target should be quantitated with an alternative ion.

### 7.2.3.1 Characteristic quantitation ions

The characteristic quantitation ions of analytes and standards are shown in the following listing. The primary quantitation ions are used (through the ID file) to produce the normal Quant report. If alternative quantitation ions are needed, use one of the secondary ions listed below. Remember, secondary ion quantitations must use the same calibration process (and files) as used for the primary ion operations.

VOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
Acetone	43	58
Acetonitrile	41	40
Acrolein	56	55,58
Acrylamide	44	71,55
Acrylonitrile	53	52,51
Allyl chloride (3-Chloro-1-propene)	76	78,41
Benzene	78	77,52
Bromobenzene	156	77,158
Bromochloromethane (target or internal std.)	128	49,130,51
Bromodichloromethane (Dichlorobromomethane)	83	85,127
4-Bromofluorobenzene (surrogate std.)	95	174,176
Bromoform	173	171,175,252
Bromomethane (Methyl bromide)	94	96,79
2-Butanone (Methyl ethyl ketone), (MEK)	43	57,72
n-Butyl alcohol (n-Butanol)	56	42,41
n-Butylbenzene	91	134
sec-Butylbenzene	105	134
tert-Butylbenzene	119	91
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	77,114
Chlorobenzene-d <sub>5</sub> (internal std.)	117	82,119
Chloroethane	64	66,49
2-Chloroethyl vinyl ether	63	65,106
Chloroform	83	85,47
Chloromethane (Methyl chloride)	50	52,49
Chloroprene (2-Chloro-1,3-butadiene)	53	88,51

VOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Dibromochloromethane (Chlorodibromomethane)	129	208,127,206
1,2-Dibromo-3-chloropropane (DBCP)	75	155,157
1,2-Dibromoethane (Ethylene dibromide), (EDB)	107	109,188
Dibromomethane (Methylene bromide)	93	95,174
1,2-Dichlorobenzene (o-Dichlorobenzene)	146	111,148
1,2-Dichlorobenzene-d <sub>4</sub>	152	115,150
1,3-Dichlorobenzene (m-Dichlorobenzene)	146	111,148
1,4-Dichlorobenzene (p-Dichlorobenzene)	146	111,148
trans-1,4-Dichloro-2-butene	75	53,89
Dichlorodifluoromethane	85	87,50,101
1,1-Dichloroethane	63	65,83,85,98
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethane-d <sub>4</sub> (surrogate std.)	65	102
1,1-Dichloroethene (1,1-Dichloroethylene)	96	61,98,63
cis-1,2-Dichloroethene (c1,2-Dichloroethylene)	96	61,98
trans-1,2-Dichloroethene (t1,2-Dichloroethylene)	96	61,98
1,2-Dichloroethene (total)	96	61,98
1,2-Dichloropropane	63	65,114,112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene (1,1-Dichloropropylene)	75	110,77
cis-1,3-Dichloropropene (c1,3-Dichloropropylene)	75	77,110
trans-1,3-Dichloropropene (t1,3-Dichloropropylene)	75	77,110
Diethyl ether (Ethyl ether)	74	59,45
1,4-Difluorobenzene (internal std.)	114	63,88
1,4-Dioxane	88	58
Ethanol (Ethyl alcohol)	45	46
Ethyl acetate	43	61,70,88
Ethylbenzene	106	91D
Ethyl methacrylate	69	41,39,99
Fluorobenzene (internal std.)	96	77
Hexachlorobutadiene	225	260
n-Hexane	86	57,43
2-Hexanone	43	58,57,100
Isobutyl alcohol	42	43,74
Isopropylbenzene (Cumene)	105	120

VOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
4-Isopropyltoluene	119	134,91
Methacrylonitrile	67	52,41
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride (Dichloromethane)	84	49,51,86
Methyl iodide (Iodomethane)	142	127,141
Methyl methacrylate	69	100
4-Methyl-2-pentanone (Methyl isobutyl ketone) (MIBK)	43	58,100
Naphthalene	128	127
Paraldehyde	45	89,117
Pentachloroethane	167	165,169,117
Propionitrile	54	55
n-Propylbenzene	91	120
Pyridine	79	52,51
Styrene	104	78,103
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	85,131,133
Tetrachloroethene (Perchloroethylene)	164	129,131,166D
Toluene (Methylbenzene)	91	92D
Toluene-d <sub>8</sub> (surrogate std.)	98	70,100
1,2,3-Trichlorobenzene	180	182
1,2,4-Trichlorobenzene	180	182
1,1,1-Trichloroethane	97	99,117,119
1,1,2-Trichloroethane	97	83D,85,99
Trichloroethene (Trichloroethylene)	130	95D,97,132
Trichlorofluoromethane (Freon 11)	101	103,66
1,2,3-Trichloropropane	75	77,110,61
1,1,2-Trichlorotrifluoroethane (Freon 113)	151	101,153,103
1,2,4-Trimethylbenzene (Pseudocumene)	105	120
1,3,5-Trimethylbenzene (Mesitylene)	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64,61
m- & p-Xylene (1,3- & 1,4-Dimethylbenzene)	106	91
o-Xylene (1,2-Dimethylbenzene)	106	91
Xylenes (total)	106	91

D signifies the primary quantitation ion for Method 524.2

#### 7.2.4 Final Output Generation

After assessing the TIC, confirming identifications (and deleting false positives), and correcting any improper integrations, a final hardcopy Quant Output report should be generated. The final report should also be automatically archived either when exiting QAREA (when no deletions are needed) or after the QDEL process by ARCV. **NOTE: At the present time only the GC/MS instruments #1 and #2 support automatic archival.** When software Revision F is available, all systems will be in communication and the remaining instruments will utilize this feature.

#### 7.3 Output Processing and Quality Assurance Checks

Data checks of the pre-run quality assurance operations should be processed as soon as the acquisition data is available. Passage or failure of the required criteria must be determined as soon as possible. Sample data should be screened in a timely manner to determine if reanalysis must be performed.

##### 7.3.1 Tune Output Processing

After acquisition of the BFB tuning solution analysis, access the total ion chromatogram and obtain the spectrum at the top of the peak. Type TUNER,=BFB and check the result to see if it passes the appropriate criteria found in paragraph 5.1.3.

If this fails, try other peak spectra or spectrum averaging to satisfy the criteria. Background subtraction can be performed (required for Method 624) if it does not distort the spectrum. **NOTE: In 1990 CLP, the mass spectrum of BFB must be acquired in the following manner: three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of BFB.**

Obtain a hard copy of the spectrum and a passing TUNER result, and file it in the GC/MS Quality Assurance Notebook for that instrument. After passing the tune criteria, run either the initial or continuing calibration. If all efforts fail to produce a spectrum which passes tuning criteria, perform manual tuning to correct the failure.

##### 7.3.2 Initial Calibration Output Processing

###### 7.3.2.1 Automated identification check

After the acquisition of one initial calibration standard, the

checking process can begin. Check the Quant Output reports to confirm that all standards analyzed were identified automatically. If any were missed, manually quantitate those compounds using program QT with the option T and determine the cause of the miss. Then use IEDIT to correct the qualifications so that all levels of the standard are recognized. Fully requantitate the standard run through QT. Apply the generic manual process to all compounds.

#### 7.3.2.2 Calibration criteria satisfaction

Update the Calibration file with the five Quant Output files using program CBUPD. Obtain a calibration report through program CBRPT with the %RSD option. Determine if the results satisfy the appropriate initial calibration criteria. If criteria are met, the analyst can continue to the next paragraph. If particular standard levels caused the failure, the analyst can reanalyze those standards only and reprocess the initial calibration as above. If criteria are still not met, suspend operations and determine whether the GC/MS system is out of control or if the standards themselves are suspect. When the problem situation is corrected, a full initial calibration may be attempted.

#### 7.3.2.3 ID file calibration

When a satisfactory initial calibration has been produced and documented, recalibrate the corresponding ID file (ID\_VCn) with the average RF's by using program CBCAL.

### 7.3.3 Continuing Calibration Output Processing

#### 7.3.3.1 Automated identification check

After the acquisition of the continuing calibration standard, review the Quant Output reports to confirm that all standards analyzed were identified automatically. If any were missed, manually quantitate those compounds using program QT with the option T and determine the cause of the miss. Then use IEDIT to correct the qualifications so that the standard is recognized. Fully requantitate the standard through QT. Apply the generic manual process to all compounds.

#### 7.3.3.2 Criteria satisfaction

Compare the continuing standard RF's to the average RF's from the initial calibration through program CBCHK, which automatically prints a report. Determine if the results satisfy the continuing calibration criteria. If criteria are met, the analyst can continue to the step in the next paragraph. If the criteria were not met, reanalyze the standard once and reprocess as above. If criteria are still not met, suspend operations and determine whether the GC/MS system is out of control or if the standard

integrity is suspect. When the problem situation is corrected, a continuing calibration may be attempted.

#### 7.3.3.3 ID file calibration

When a satisfactory continuing calibration has been produced and documented, recalibrate the daily ID file (ID\_VDn) with the continuing standard RF's by using program QCAL. Since this program also sets retention time windows based on the daily standard, the standard identifications must be correct!

#### 7.3.4 Laboratory Blank Output Processing and Criteria

##### 7.3.4.1 Standards identification check

If any standard (internal or surrogate) is not automatically identified by the ID file, then the ID file should be edited through IEDIT so that every standard is identified. Requantitate the laboratory blank through the corrected ID file using program QT.

##### 7.3.4.2 Standards abundance check

The Quant report should be reviewed to check the internal and surrogate standards for satisfaction of the criteria found in paragraphs 7.3.6 or 7.3.7. If **any** fail the required criteria, immediately enter QAREA and correct the integration. If the failure cannot be corrected, reanalyze immediately. If a problem still exists, correct it before any more runs are acquired.

##### 7.3.4.3 Blank target values check

Perform the generic manual validation procedure. The target analyte concentrations in the laboratory blank must be below certain level to show the absence of contamination. The required criteria is based on the 1990 CLP Statement of Work (except for the differing criteria for Method 524.2).

The laboratory blank must contain less than or equal to five times the quantitation limit of methylene chloride, acetone and 2-butanone. For all other target compounds not listed above, the method blank must contain less than or equal to the quantitation limit of any single analyte. For Method 524.2, background from method analytes should be below the method detection limit.

### 7.3.5 Sample Output Processing

#### 7.3.5.1 Standards identification check

If any standard (internal or surrogate) is not automatically identified by the ID file, then the ID file should be edited through IEDIT so that every standard is identified. Requantitate the sample through the corrected ID file using program QT.

#### 7.3.5.2 Standards abundance check

The Quant report should be reviewed to check the internal and surrogate standards for satisfaction of the criteria found in paragraphs 7.3.6 or 7.3.7. If any fail the required criteria, immediately enter QAREA and correct the integration. If the failure cannot be corrected, reanalyze at the analyst's convenience. If the problem persists for many samples, make a new fortification standard, run a new calibration or start the whole shift from the beginning.

#### 7.3.5.3 Sample validation

Perform the generic manual verification procedure. If the concentration of any analyte exceeds the initial calibration range, that sample must be diluted and reanalyzed so that all targets are within the initial calibration range.

#### 7.3.5.4 Blanks and spikes

Other blanks (field, trip, holding and laboratory fortified) along with the matrix spike and matrix spike duplicate should be processed as a sample.

#### 7.3.5.5 Tentatively identified compound processing

When requested, perform a search of the data file for the ten highest non-target peaks that have abundances above 10% of the abundance of the nearest (by retention time) internal standard. These are identified (if possible), semi-quantitated, and referred to as tentatively identified compounds (TIC's). Invoke program FTIC by entering: ,\*FTICV, [Data File Namr]. This generates the raw data needed to process TIC's.

Be cautious when utilizing identifications produced by automatic probability matching to the spectral data base. Proper interpretation considers automatic and manual spectral matching, retention time, probability of occurrence and chemical knowledge to tentatively identify unknown components. Unless the analyst is certain of the correct identification, interpretation should be performed by the GC/MS Supervisor or the Organics Group Leader. Semi-quantitation is automatically performed by FTIC except the final concentration estimates involving soils are computed incorrectly. Perform manual calculation to rectify the

error in the correction for percent moisture.

### 7.3.6 Surrogate Standard Quality Assurance Criteria

Surrogate standards are added to every sample, blank and spike. The criteria for acceptance are as follows:

7.3.6.1 Surrogate criteria for Methods 624 and 524.2  
Specific criteria for each surrogate are not listed for these methods. However, the responses should not vary more than 50% over the course of a shift. If the response during an acquisition varies more than this, the GC/MS system is out of control and the analysis should be performed again.

7.3.6.2 Surrogate criteria for 8260 and CLP methodologies  
The following table lists the required surrogate spike recovery limits in percent of amount spiked:

Surrogate Compound	Water	8260/1988 CLP Soil/Sediment	1990 CLP Soil/Sediment
1,2-Dichloroethane-d <sub>4</sub>	76-114	70-121	70-121
Toluene-d <sub>8</sub>	88-110	81-117	84-138
4-Bromofluorobenzene	86-115	74-121	59-113

### 7.3.7 Internal Standard Quality Assurance Criteria

Internal standard responses and retention times must be evaluated after the data acquisition.

7.3.7.1 Retention time criteria  
If the retention time for any internal standard changes by more than thirty seconds, the chromatographic system must be inspected for malfunctions, and corrections made as required. Note that very large compound responses indicate column overloading leading to chromatographic aberrations and can cause retention time shifting.

7.3.7.2 Response criteria  
If the extracted ion current profile area (quantitation ion integration) for any internal standard changes by more than a factor of two (-50% to 100%), from the latest daily calibration standard, the analysis should be rerun. If the problem continues with reanalysis or other analyses, the GC/MS system must be inspected for malfunction, and corrections made as appropriate.

The Method 524.2 internal standard response limits are -50% to 150%.

### 7.3.8 Matrix Spike Quality Assurance Criteria

The matrix spikes and spike duplicates are used to evaluate the matrix effect of a sample upon the analytical methodology. Other than for Method 524.2, the criteria are only advisory and are used to indicate when sample matrix effects occur.

7.3.8.1 Method 524.2 fortification sample and blank requirements  
Method 524.2 uses fortification blanks and fortified samples for matrix evaluations (and to determine if established detection limits can be met). All target analytes are spiked before extraction and analyzed as samples.

Calculate the measured concentration and accuracy of each analyte. For each analyte, the accuracy (expressed as a percentage of the true value) should be 80-120%. Some analytes, mostly early eluting gases and late eluting higher molecular weight compounds, are measured with less accuracy. If acceptable accuracy and detection limits cannot be achieved, the problem must be located and corrected before further samples are analyzed.

### 7.3.8.2 Method 8260 and CLP matrix spike / spike duplicate criteria

The matrix spike and matrix spike duplicate recovery (%R) target limits for each spike compound are listed below. The targets for the maximum relative percent difference (RPD) between the recoveries of the matrix spike and the matrix spike duplicate are also listed.

Matrix Spike Compound	%R		RPD	
	Water	Water	Soil	Soil
1,1-Dichloroethane	61-145	14	59-172	22
Trichloroethene	71-120	14	62-137	24
Benzene	76-127	11	66-172	21
Toluene	76-125	13	59-139	21
Chlorobenzene	75-130	13	60-133	21

## 8.0 SYSTEM MAINTENANCE

### 8.1 Carrier/Purge Gas Supply Line

The oxygen/moisture trap in the helium supply line should be

replaced at least once per year or when indicated. The hydrocarbon trap within the main purge and trap unit should be replaced at least every two years or when indicated.

## 8.2 Instrumentation

### 8.2.1 Purge and Trap Maintenance

#### 8.2.1.1 Sparge tubes

Sparge tubes are removed and cleaned after every use. After the cleaning procedures below are completed, all spargers are oven dried at 105° C for at least 1 hour.

Fritted tubes which contained large amounts of organic purgeables are backrinsed three times with hot tap water followed by three backrinses with deionized water. These spargers along with all other used fritted tubes are backflushed with Milli-Q water as the final process.

The inside of all used soil (needle) spargers is scrubbed using a bristled brush and hot tap water. The tubes are then thoroughly rinsed three times with deionized water, and twice with Milli-Q water.

#### 8.2.1.2 Automatic sampler lines

The lines from a particular sparge port and the heated transfer (auxiliary) line between the automatic sampler and the purge and trap main unit should be cleaned whenever a sample with extremely high contamination is purged or water enters the lines. If neither occurs within a three month period, perform the cleaning as periodic maintenance.

Remove the auxiliary transfer line outlet from the purge and trap main unit and reduce the line's temperature setpoint to ambient. Clean the automatic sampler lines by purging 0.5 mL of purge-and-trap methanol through the contaminated port (or all port if scheduled cleaning is being performed) for 20 minutes. Following methanol purging, increase the auxiliary transfer line temperature to 105° C and purge reagent water in the same port(s) for at least one hour. **NOTE: Do not allow the purge and trap unit to operate in the DESORB mode during the above operations!**

Reset the temperature and reconnect the transfer line before attempting normal operations.

#### 8.2.1.3 Main purge and trap unit

The purge and trap main unit should be "baked out" at least every week or whenever a very highly contaminated sample has been

purged. The bake-out procedure involves setting the unit to a constant bake cycle and raising temperature setpoints overnight. The bake-out setpoints are 105° C for the regular transfer line and 150° C for the valve.

Every three months, clean the transfer line between the unit and the gas chromatograph by the following method. Turn off the mass spectrometer electronics and after at least 45 minutes, turn off the MS pumps. Reduce the transfer line temperature to ambient. Remove the end of the transfer line connected to the injection port (or the GC column). Remove the main unit end of the transfer line from the valve (or the anhydrator) and connect this 1/16" line with a female-female coupler to another 1/16" line with a luer-lock opposite end. Fill a 5 mL luer-lock syringe with purge-and-trap methanol and connect it to the luer-lock connection on the main unit end of the transfer line. Slowly inject the methanol through the transfer line and collect the effluent into a beaker for disposal.

Reconnect the end of the transfer line to the main unit. Increase the setpoint temperature of the line to 105° C and let carrier gas flow through the transfer line overnight. The next day, connect the GC side of the transfer line and reset temperature setpoints to normal.

The trap should be replaced if it is indicated that water has saturated into it. The trap should also be replaced when a reduced recovery of brominated compounds is observed.

#### 8.2.1.4 Anhydrator regeneration

The anhydrator, over a period of time, can become saturated with water, methanol or organics. The device should be removed and regenerated at least once every six months. First flush with 1mL purge-and-trap methanol. Attach a source of nitrogen to the inlet end of the anhydrator and flush for a minimum of one hour at 100 to 200 mL/min.

### 8.2.2 Gas Chromatographic System Maintenance

#### 8.2.2.1 Gas chromatographic column

The GC column should be altered or replaced when degradation of the chromatography approaches an unacceptable level. Chromatographic degradation can be indicated by poorly shaped peaks, grossly tailing peaks, unsatisfactory quality assurance results or loss of retention.

Column "clipping" can be used to alter the column and prolong

usage. It is performed by cutting off 18 to 36 inches of the front of the column with a column cutting tool. The system, of course, should be shut down during this procedure. Column rinsing with solvents is not recommended to regenerate columns.

### 8.2.3 Mass Spectrometer Maintenance

#### 8.2.3.1 Ion Source cleaning

A dirty source is indicated by loss of overall sensitivity, difficulty in instrumental tuning and, most indicatively, a loss of high mass sensitivity. The source should be removed, disassembled, cleaned, rinsed carefully, reassembled and installed. The lens stack in the source should be disassembled for cleaning **only** when there is very noticeable discoloration or every two years. The ceramic source parts should be heated to 400° C with every other cleaning and soaked in aqua regia (with subsequent rinsing) once every two years or when current can be carried across a ceramic source part. Nylon gloves are worn during all operations after a rinsing step.

#### 8.2.3.2 Filament replacement

A total loss of signal indicates a broken filament. Switch to the alternate filament if available. If both filaments are broken, they should both be replaced. If the source is removed for cleaning and one filament is broken, it should be replaced at that time. Broken filaments should be accumulated until only one spare is left (per instrument) and then sent for repair instead of purchasing new replacement.

#### 8.2.3.3 Quadrupole rod cleaning

The quadrupole rods should be cleaned when dark ion burns are noted near on the rods. **Do not disassemble the rod cluster.** Clean the burn marks with a series of organic solvents using a cotton applicator, then soak the dirty end in solvent overnight.

### 8.3 Data System

#### 8.3.1 File Maintenance

##### 8.3.1.1 File purging

Unneeded file should be purged to make room for acquisitions. Data files should be reviewed monthly through SHODA after logging on with MANAGER. Any files older than one month which are through processing and have been reported, should be purged.

Quant output files accumulate on the Aquarius default cartridge and should be reviewed every two months and purged when no

further utilization is apparent.

Both types of archival operations should be completed before file purging.

#### 8.3.1.2 Cartridge packing

Cartridge packing should be performed after any significant file purging. The first cartridge on each disc's cartridge list (QT) should be packed weekly since system operations create and purge files on this disc invisibly.

#### 8.3.2 Manual File Archiving

In addition to automatic Aquarius archiving, Kiber Analytical produces backup archival tapes using the streaming process. These operations should be performed monthly before file purging operations are performed.

## GC/MS SEMIVOLATILES ANALYSIS

### 1.0 SUMMARY OF PROCEDURE

This procedure is used for the GC/MS analysis of semivolatile extracts, and for confirmation of pesticides/PCB's identified by GC/EC, if concentrations permit. An aliquot of the sample is injected onto the analytical column and the gas chromatograph is temperature programmed to separate the target analytes. These are then detected with a quadrupole mass spectrometer interfaced to a data system used for processing and archiving data.

### 2.0 APPARATUS, REAGENTS AND SUPPLIES

#### 2.1 Apparatus

##### 2.1.1 Balance

An analytical balance capable of accurately weighing +/- 0.0001 g is used for preparing standards.

##### 2.1.2 Nitrogen evaporator

A nitrogen evaporation device is used to evaporate small volumes of extraction solvents for subsequent dilution to volume for final extracts.

##### 2.1.3 GC/MS System

The GC/MS system consists of an automatic sampler, automatic injector, gas chromatograph, analytical column, mass spectrometer, data system and archival system.

##### 2.1.3.1 Gas chromatograph

The gas chromatographic system must be capable of accurate temperature programming and have a flow controller that maintains a constant column flow rate throughout the temperature program

operations. An automatic sampler and automatic injector should be interfaced to the gas chromatograph for efficiency and reproducibility. All GC carrier gas lines are constructed from solvent-cleaned stainless steel tubing, thoroughly dried before utilization.

#### 2.1.3.2 Analytical column

A 30 m long x 0.25 mm ID DB-5.625 fused silica capillary column with a 0.5  $\mu$ m film thickness is used. The column inlet is connected to the injector port for splitless injection and the column outlet is directly interfaced to the mass spectrometer so that the end of the column resides 2 mm from the source repeller.

#### 2.1.3.3 Mass spectrometer

A quadrupole mass spectrometer utilizing 70 volts (nominal) electron energy in the electron impact ionization mode is used. It must be capable of scanning from 35 to 500 amu every 1 second or less and capable of producing a mass spectrum which meets the appropriate tuning criteria when 50 ng of DFTPP (decafluorotriphenylphosphine) is injected through the GC inlet. The instrument vacuum pumps have an in-line carbon trap collecting the organic effluent to prevent the release of untrapped contaminants.

#### 2.1.3.4 Data system

A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage, on machine readable media, of all mass spectra obtained throughout the duration of the chromatographic program. The computer has Aquarius software which allows automated searching of any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This software allows integration of the abundance of any such plot between specified time or scan number limits. This software allows for the comparison of sample spectra of unknowns against reference library spectra in the 1989 release of the NIST/EPA/MSDC mass spectral library. The data system must be capable of flagging all data files that have been edited manually by laboratory personnel.

#### 2.1.3.5 Archival system

The data system must contain a streaming magnetic tape storage device suitable for long-term, off-line storage.

## 2.2 Reagents and Supplies

### 2.2.1 Reagents

#### 2.2.1.1 Methylene chloride

The methylene chloride used to finalize extracts is pesticide grade or purer.

#### 2.2.1.2 Sodium bicarbonate

Powdered, anhydrous sodium bicarbonate (ACS) is used to neutralize extracts from TCLP extraction procedures. Further anhydrate and purify by heating at 400° C for four hours, cooling in a dessicator and storing in a glass bottle.

#### 2.2.1.3 Sodium sulfate

Powdered, anhydrous sodium sulfate (ACS) is used to remove water after sodium bicarbonate neutralization. Further anhydrate and purify by heating at 400° C for four hours, cooling in a dessicator and storing in a glass bottle.

#### 2.2.1.4 Gas supply

The carrier gas is zero grade helium equipped with an in-line trap for the removal of moisture and oxygen.

### 2.2.2 Supplies

#### 2.2.2.1 Micro Dispensers

Positive displacement capillary dispensers are used to measure microliter amounts of standard solutions. They should have the ability to dispense variable amounts, with maximum volumes of 25 and 100 uL.

#### 2.2.2.2 Glassware

##### 2.2.2.2.1 Volumetric glassware

1, 2, 5, and 10mL Class A volumetric flasks are used for final extract dilutions. Glassware utilized must be clean and at ambient temperature.

##### 2.2.2.2.2 Disposable pasteur pipettes

Disposable glass pipettes are used for sodium bicarbonate and sodium sulfate cleanups.

##### 2.2.2.2.3 Vials

1.8 and 5 mL screw-cap vials are used for long term storage of sample extracts. The cap must be teflon lined.

#### 2.2.2.2.4 Automatic GC sampler vials, crimp-top

#### 2.2.2.3 Glass wool

Solvent (methylene chloride) cleaned glass wool is used to hold reagents for cleanup of TCLP extracts.

### 3.0 STORAGE AND PREPARATION

#### 3.1 Extract Storage

Sample extracts are stored in the extract refrigerator at 4 +/- 2° C in the Sample Control area under the control of the Sample Custodian. Extracts of samples are signed out for GC/MS analysis on the Internal Chain of Custody Form by the analyst, who is responsible for the custody of those extracts until returned to the Sample Custodian and signed back in on the Internal Chain of Custody Form. Extracts of finished samples are held in the extract refrigerator for 3 months.

#### 3.2 Analytical Standard Storage

All analytical standards in solution are stored in teflon-lined screw-cap vials in a discrete freezer at -10° C to -20° C located in the Sample Control room, along with neat standards. Alternative storage procedures can be used if these conditions are detrimental to the integrity of the standard or unsafe. Each standard or solution must be discretely designated and entered into the Standards Notebook along with all preparation operations and/or manufacturer's documentation.

#### 3.3 Analytical Standard Preparation

##### 3.3.1 Target Calibration Standards

##### 3.3.1.1 Stock standard solutions

Stock standard solutions may be prepared from standard materials or purchased. Prepare the solutions, using assayed EPA traceable standards at ambient temperature, by weighing a known amount into a volumetric flask and diluting to volume with methylene chloride (or other solvent if necessary). **Store in a tightly closed vial in a freezer**, unless in use. During use, leave the standard containers open for the shortest time possible and return to the freezer as soon as operations are complete.

### 3.3.1.2 Working calibration standards

Normal working standard levels are at 20, 50, 80, 120 and 160 ng/uL (0.1, 0.5, 1, 2, 5, and 10 ng/uL for Method 525 targets) for a 1 uL injection. The 50 ng/uL (525: 5 ng/uL) standard is used as the continuing calibration check standard. For Method 525, pentachlorophenol standard concentration levels should be 4X the normal levels listed above and toxaphene should be 12.5X.

A duplicate set of working calibration standards should be created and stored along with four extra continuing calibration check standards. These extra standards are used when degradation of sensitive compounds or leaching of phthalate esters occurs.

Use the micro-dispenser to take an aliquot of the stock standard solution (at ambient temperature) and deposit into a volumetric flask. Additional standards and/or stock solutions of other target analytes may be added to the flask if no reaction or degradation of targets occur. DFTPP for tuning is added to the 50 ng/uL standard if following the 1990 CLP SOW. Add a volume of the internal standard spiking solution to bring their final levels to 40 ng/uL (525: 5 ng/uL). Bring to volume with methylene chloride while mixing. Transfer the standard solution to a screw-capped vial and store as above, unless in use. During use, leave the standard containers open for the shortest time possible. Fresh standards should be prepared every twelve months, at a minimum.

### 3.3.2 Internal Standards

The internal standards used are 1,4-dichlorobenzene-d<sub>4</sub>, naphthalene-d<sub>8</sub>, acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub>, chrysene-d<sub>12</sub> and perylene-d<sub>12</sub>. Method 525 utilizes only acenaphthene-d<sub>10</sub>, phenanthrene-d<sub>10</sub> and chrysene-d<sub>12</sub>.

#### 3.3.2.1 Internal standard spiking solution

A commercially prepared mixture of standards in a 2000 ug/mL methylene chloride solution is used as the internal standard spiking solution for addition to target standard solutions and sample extracts (except for Method 525, which are added before extraction). Each internal standard purity must be 99% or better.

### 3.3.3 Surrogate Standards

Method 525 utilizes perylene-d<sub>12</sub> as the surrogate standard. Phenol-d<sub>5</sub>, 2-fluorophenol and 2,4,6-tribromophenol are used for acid extracts and base/neutral extracts use nitrobenzene-d<sub>5</sub>, 2-fluorobiphenyl and p-terphenyl-d<sub>14</sub>. The surrogate standards for combined extracts consists of all of those used for the acid and base/neutral extracts. In addition to these surrogates, the 1990 CLP SOW uses two extra compounds with advisory limits, 2-Chlorophenol-d<sub>4</sub> and 1,2-Dichlorobenzene-d<sub>4</sub>.

#### 3.3.3.1 Stock surrogate standard solutions

Stock surrogate standard solutions (except for Method 525) are prepared from neat standards of 99% purity or better. Prepare stock surrogate solutions, using assayed standards at ambient temperature, by weighing a known amount into a volumetric flask and diluting to volume with methylene chloride. **Keep all standards tightly closed and in the freezer at all times**, unless in use. During use, leave the standard containers open for the shortest time possible. The concentrations are 2000 ug/mL for the acid surrogates and 1000 ug/mL for the base/neutral surrogates. The **same** solutions are used to prepare surrogate standard calibration solutions **and** in the extraction procedure. The semivolatile analyst is responsible for the stock surrogate standard solution preparations.

#### 3.3.3.2 Surrogate standard calibration solutions

Surrogate standard calibration solutions are prepared as target calibration solutions following the procedures in paragraph 3.3.1.2.

### 3.3.4 Tuning Check Compound

Decafluorotriphenylphosphine (DFTPP) is the semivolatile instrument performance check compound.

#### 3.3.4.1 Tuning check solution

Prepare a 50 ng/uL solution of DFTPP and store it in a freshly-capped autoinjector vial. It can be added to the daily calibration standard check solution at 50 ng/uL.

#### 3.3.4.2 Column conditioning solution

Prepare a 5 ug/uL solution of DFTPP and store it in a capped injector vial. One microliter of this solution is injected into the GC daily to precondition the analytical column.

### 3.3.5 Matrix Spike Compounds

The matrix spiking compounds for all methodologies (except 525) are 1,2,4-trichlorobenzene, acenaphthene, 2,4-dinitrotoluene, pyrene, N-nitroso-di-n-propylamine and 1,4-dichlorobenzene for base/neutral extracts and phenol, pentachlorophenol, 2-chlorophenol, 4-chloro-3-methylphenol and 4-nitrophenol for acid extracts. Full analyses use all the acid and base/neutral matrix spiking compounds. Method 525 utilizes a fortification sample which has been spiked with all target analytes. These matrix spiking solutions are prepared and used in the extraction procedure.

### 3.4 Final Extract Preparation

The GC/MS analyst obtains the extract after all extraction, screening and cleanup procedures have been employed. All extracts are allowed to reach ambient temperature before diluting to final volume.

#### 3.4.1 Non-TCLP Extracts

##### 3.4.1.1 Undiluted samples

Samples requiring no dilution are prepared using all of the extract. Using a glass pasteur pipette, quantitatively transfer the extract to a 1 mL volumetric flask. Use a small amount of reagent methylene chloride to rinse the extract vial and add this to the volumetric flask, using (and rinsing) the initial transfer pipette with it. Add 20 uL of the internal standard spiking solution with a capillary micro-dispenser. Bring to volume with reagent methylene chloride. Mix the flask contents by using a clean pipette, drawing the solution up into the pipette and then expelling it back into the flask. Repeat this mixing procedure twice more, then transfer the solution to a clean autosampler vial, put on a cap, and crimp it. The crimp cap should be tight enough so that it cannot be rotated, but not so tight that it is deformed.

##### 3.4.1.2 Diluted samples

Samples requiring dilution, are first diluted with methylene chloride, using volumetric glassware, to the proper dilution. Add approximately 0.8 mL of the diluted extract to a fresh 1 mL volumetric flask. Add 20 uL of the internal standard spiking solution to the flask and then bring to volume with additional diluted extract. Mix the flask contents by using a clean

disposable pasteur pipette, drawing the solution up into the pipette and then expelling it back into the flask. Repeat this mixing procedure twice more, then transfer the solution to a clean autosampler vial and cap it. The cap should be tight enough so that it cannot be rotated, but not so tight that it is deformed.

#### 3.4.2 TCLP Extracts

Test the pH of the extract after taking 10 uL, diluting to 1 mL with distilled water, mixing and letting stand for 5 minutes. If the pH is at or above 6.0, proceed with the normal extract preparation above. If the extract has a pH below 6.0, use the following cleanup procedure as many times as necessary.

##### 3.4.2.1 Sodium bicarbonate neutralization

Prepare a neutralization microcolumn by inserting glass wool into a pasteur pipette and lodging it firmly in the bottom neck. Add sodium bicarbonate to create a 4 cm depth. With a clean pipette, quantitatively transfer the extract (of a TCLP extract) onto the microcolumn pipette and collect the eluate in a vial. Moderate pressure may be applied with a pipette bulb to accelerate the flow through the microcolumn. Rinse the original extract vial with fresh methylene chloride using (and rinsing) the original transfer pipette and put this rinsate through the microcolumn also. **Do not allow the sodium bicarbonate packing to go to dryness.**

##### 3.4.2.2 Sodium sulfate drying

Following sodium bicarbonate neutralization, dry the collected eluate through a microcolumn of sodium sulfate prepared as above. After collection of all column outputs into a vial, reduce the volume to less than 1 mL with the nitrogen evaporator. Retest the pH and either neutralize again if it is too acidic or proceed from here through the normal extract preparation procedure.

## 4.0 INSTRUMENT OPERATING CONDITIONS

### 4.1 Gas Chromatograph

Recommended gas chromatographic analytical conditions:

Carrier gas:	helium
Flow rate:	1.1 mL/min
Injection port temperature:	270° C
MS Interface temperatures:	280° C
Initial temperature:	60° C
Initial hold time:	4 minutes
Temperature ramp rate:	8° C/minute
Final temperature:	320° C
Final hold time:	5.5 mins or until all targets elute

### 4.2 Mass Spectrometer

Required mass spectrometric analytical conditions:

Electron energy:	70 volts (nominal)
Mass range:	35-500 amu
Scan time:	not to exceed 1 second per scan
Injection purge gas delay:	0.8 minutes

## 5.0 CALIBRATION

### 5.1 Instrument Performance Calibration

Prior to the analysis of any samples, blanks or standards, it must be established that the GC/MS system meets the specified ion abundance criteria for the tuning check solution.

#### 5.1.1 Manufacturer's Internal Calibration Compound

Neat PFTBA (perfluorotributylamine) is used to monitor the MS source parameters when tuning. It is introduced through a valved induction tube, by vaporization directly into the source chamber.

#### 5.1.2 System Performance Calibration Compound

DFTPP is introduced by analyzing the tuning check solution (3.3.4.1) or a daily calibration check standard which contains DFTPP, as a sample. Gas chromatographic temperature programming alterations are allowed to expedite the procedure.

### 5.1.3 Ion abundance criteria

There are three different criteria for DFTPP tuning. Traditional criteria are used for Methods 625 and 8270 and the 1988(1989) CLP protocol. The 1990 CLP SOW and Method 525 each have different criteria. This laboratory follows the traditional criteria except when required or requested otherwise. A mass spectrum from the analysis of the tuning solution must meet the following ion abundance criteria.

#### 5.1.3.1 Traditional tuning criteria

Mass	Ion Abundance Criteria
51	30.0 - 60.0 percent of mass 198
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	40.0 - 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0 - 23.0 percent of mass 442

#### 5.1.3.2 Method 525 tuning criteria

The peak spectra may be averaged for Method 525.

Mass	Ion Abundance Criteria
51	10.0 - 80.0 percent of the base peak
68	less than 2.0 percent of mass 69
70	less than 2.0 percent of mass 69
127	10.0 - 80.0 percent of the base peak
197	less than 2.0 percent of mass 198
198	base peak or greater than 50.0 percent of mass 442
199	5.0 - 9.0 percent of mass 198
275	10.0 - 60.0 percent of the base peak
365	greater than 1.00 percent of the base peak
441	present but less than mass 443
442	base peak or greater than 50.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

### 5.1.3.3 New CLP (1990) tuning criteria

The mass spectrum of DFTPP must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of DFTPP.

#### Mass Ion Abundance Criteria

---

51	30.0 - 80.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	25.0 - 75.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent relative abundance (see note)
199	5.0 - 9.0 percent of mass 198
275	10.0 - 30.0 percent of mass 198
365	greater than 0.75 percent of mass 198
441	present but less than mass 443
442	40.0 - 110.0 percent of mass 198
443	15.0 - 24.0 percent of mass 442

Note: All ion abundances **must** be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may be up to 110 percent that of m/z 198.

## 5.2 Initial Calibration

Prior to the analysis of samples and blanks, and after the tuning criteria have been met, each GC/MS system must be initially calibrated to determine instrument sensitivity and the linearity of response.

### 5.2.1 Calibration Standard Analyses

Analyze five (six for Method 525) concentrations of target analytes following the preparations in paragraph 3.3.1.2 and the operating conditions and procedures in paragraphs 4.0 and 6.3. Process the initial calibration (name sequentially in format shown in overall GC/MS procedure) through CBUPD and CBRPT which automatically calculates and reports the relative response factors (RRF), average response factors and the percent relative standard deviations (%RSD) using the formulas outlined in the following paragraphs.

### 5.2.2 Relative Response Factor Calculation

Tabulate the area response of the characteristic ions in the extracted ion current profile against concentration for each compound and internal standard and calculate relative response factors (RRF) for each compound using the following equation:

$$\text{RRF} = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

$A_x$  = Area of the characteristic ion for the target compound  
 $A_{is}$  = Area of the characteristic ion (EICP) for the specific internal standard  
 $C_{is}$  = Concentration of the internal standard  
 $C_x$  = Concentration of the target compound

### 5.2.3 Average RRF and Relative Standard Deviation Calculation

The average relative response factor ( $\overline{\text{RRF}}$ ) must be calculated for all compounds. Calculate the % Relative Standard Deviation (%RSD) of RRF values over the working range of the curve.

$$\% \text{RSD} = \frac{\text{Standard deviation}}{\text{mean}} \times 100$$

### 5.2.4 Initial Calibration Response Factor Criteria

Response factor criteria are used for semivolatile target compounds and surrogate standards. The variant criteria which exist for Methods 625, 8270, 525 and the CLP Statements of Work (SOW) issued in 1988 and 1990 are listed in the paragraphs below. The 1988 CLP SOW criteria are used unless otherwise specified.

#### 5.2.4.1 Method 625 response factor criteria

The RSD of all analytes and surrogates must be < or = to 35%.

#### 5.2.4.2 Method 8270 response factor criteria

The Method 8270 criteria are identical to the 1988 CLP criteria.

### 5.2.4.3 Method 525 response factor criteria

The RSD of all analytes and surrogates must be less than or equal to 30%. As an alternative to calculating mean RF's and applying the %RSD criteria, the Aquarius software can be utilized to generate a second or third order regression calibration curve.

### 5.2.4.4 CLP response factor criteria

The compounds listed below must satisfy the minimum RRF and maximum %RSD criteria for the semivolatiles initial calibration. Note that the 1990 CLP Statement Of Work allows four compounds (with an RRF >.010 requirement and a specified %RSD) to be noncompliant. However, the RRF's for those four compounds must be greater than or equal to .010, and the %RSD must be less than or equal to 40.0 for the initial calibration to be considered acceptable.

Semivolatiles Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
Phenol		.800	30.0	20.5
bis(2-Chloroethyl) ether		.700		20.5
2-Chlorophenol		.800		20.5
1,3-Dichlorobenzene		.600		20.5
1,4-Dichlorobenzene		.500	30.0	20.5
1,2-Dichlorobenzene		.400		20.5
2-Methylphenol		.700		20.5
bis(2-Chloroisopropyl) ether / 2,2'-oxybis(1-Chloropropane)		.010		
4-Methylphenol		.600		20.5
N-Nitroso-di-n-propylamine	.050	.500		20.5
Hexachloroethane		.300		20.5
Nitrobenzene		.200		20.5
Isophorone		.400		20.5
2-Nitrophenol		.100	30.0	20.5
2,4-Dimethylphenol		.200		20.5
bis(2-Chloroethoxy) methane		.300		20.5
2,4-Dichlorophenol		.200	30.0	20.5
1,2,4-Trichlorobenzene		.200		20.5
Naphthalene		.700		20.5
4-Chloroaniline		.010		
Hexachlorobutadiene		.010	30.0	
4-Chloro-3-methylphenol		.200	30.0	20.5
2-Methylnaphthalene		.400		20.5
Hexachlorocyclopentadiene	.050	.010		
2,4,6-Trichlorophenol		.200	30.0	20.5
2,4,5-Trichlorophenol		.200		20.5
2-Chloronaphthalene		.800		20.5
2-Nitroaniline		.010		

Semivolatile Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
Dimethyl phthalate		.010		
Acenaphthylene		1.300		20.5
3-Nitroaniline		.010		
Acenaphthene		.800	30.0	20.5
2,4-Dinitrophenol	.050	.010		
4-Nitrophenol	.050	.010		
Dibenzofuran		.800		20.5
2,4-Dinitrotoluene		.200		20.5
2,6-Dinitrotoluene		.200		20.5
Diethylphthalate		.010		
4-Chlorophenyl-phenylether		.400		20.5
Fluorene		.900		20.5
4-Nitroaniline		.010		
4,6-Dinitro-2-methylphenol		.010		
N-Nitrosodiphenylamine		.010	30.0	
4-Bromophenyl-phenylether		.100		20.5
Hexachlorobenzene		.100		20.5
Pentachlorophenol		.050	30.0	20.5
Phenanthrene		.700		20.5
Anthracene		.700		20.5
Carbazole		.010		
Di-n-butylphthalate		.010		
Fluoranthene		.600	30.0	20.5
Pyrene		.600		20.5
Butylbenzylphthalate		.010		
3,3'-Dichlorobenzidene		.010		
Benzo(a)anthracene		.800		20.5
bis(2-Ethylhexyl)phthalate		.010		
Chrysene		.700		20.5
Di-n-octyl phthalate		.010	30.0	
Benzo(b)fluoranthene		.700		20.5
Benzo(k)fluoranthene		.700		20.5
Benzo(a)pyrene		.700	30.0	20.5
Indeno(1,2,3-cd)pyrene		.500		20.5
Dibenz(a,h)anthracene		.400		20.5
Benzo(g,h,i)perylene		.500		20.5
Nitrobenzene-d <sub>5</sub>		.200		20.5
2-Fluorobiphenyl		.700		20.5
Terphenyl-d <sub>14</sub>		.500		20.5
Phenol-d <sub>5</sub>		.800		20.5
2-Fluorophenol		.600		20.5
2,4,6-Tribromophenol		.010		
2-Chlorophenol-d <sub>4</sub>		.800		20.5
1,2-Dichlorobenzene-d <sub>4</sub>		.400		20.5

### 5.3 Continuing Calibration

Prior to the analysis of samples and after daily tuning criteria have been met, a calibration check (against the initial calibration) must be performed once every working shift on each GC/MS system, unless an initial calibration was performed.

#### 5.3.1 Calibration Standard Analysis

Analyze a continuing check standard of target analytes using operations in paragraphs 3.3.1.2, 4.0 and 6.3. Process the continuing calibration through CBCHK, which calculates the continuing RRF's and percent difference (%Diff) between them and the average RRF's generated by the initial calibration.

#### 5.3.2 Continuing Calibration Response Factor Criteria

The continuing calibration criteria are described below. Check the RRF's of those compounds for which minimum values have been established. If these criteria are met, the RRF's for all compounds are calculated and reported. Compare the %Diff with any criteria listed in the paragraphs below. The 1988 CLP criteria are used unless otherwise specified.

##### 5.3.2.1 Method 625 calibration curve verification criteria

The working calibration curve must be verified on each working day by the measurement of a calibration standard. If the response for any parameter varies more than +/- 20%, the test must be repeated using a fresh calibration standard or a new calibration curve must be prepared for the compound(s).

##### 5.3.2.2 Method 8270 continuing response factor criteria

These criteria are identical to the 1988 CLP criteria.

##### 5.3.2.3 Method 525 continuing response factor criteria

The response factor for each analyte and surrogate must be within 30% of the mean value measured in the initial calibration. If a second or third order regression is used, the point from the check standard for each analyte and surrogate must fall, in the analyst's judgement, on the initial calibration curve.

##### 5.3.2.4 CLP calibration check criteria

The compounds with entries listed below must meet the minimum RRF criteria and the maximum %Diff criteria for the continuing calibration to be considered acceptable.

Semivolatile Compound	Minimum RRF		Maximum %Diff	
	1988	1990	1988	1990
Phenol		.800	25.0	25.0
bis(2-Chloroethyl)ether		.700		25.0
2-Chlorophenol		.800		25.0
1,3-Dichlorobenzene		.600		25.0
1,4-Dichlorobenzene		.500	25.0	25.0
1,2-Dichlorobenzene		.400		25.0
2-Methylphenol		.700		25.0
bis(2-Chloroisopropyl)ether / 2,2'-oxybis(1-Chloropropane)		.010		
4-Methylphenol		.600		25.0
N-Nitroso-di-n-propylamine	.050	.500		25.0
Hexachloroethane		.300		25.0
Nitrobenzene		.200		25.0
Isophorone		.400		25.0
2-Nitrophenol		.100	25.0	25.0
2,4-Dimethylphenol		.200		25.0
bis(2-Chloroethoxy)methane		.300		25.0
2,4-Dichlorophenol		.200	25.0	25.0
1,2,4-Trichlorobenzene		.200		25.0
Naphthalene		.700		25.0
4-Chloroaniline		.010		
Hexachlorobutadiene		.010	25.0	
4-Chloro-3-methylphenol		.200	25.0	25.0
2-Methylnaphthalene		.400		25.0
Hexachlorocyclopentadiene	.050	.010		
2,4,6-Trichlorophenol		.200	25.0	25.0
2,4,5-Trichlorophenol		.200		25.0
2-Chloronaphthalene		.800		25.0
2-Nitroaniline		.010		
Dimethyl phthalate		.010		
Acenaphthylene		1.300		25.0
3-Nitroaniline		.010		
Acenaphthene		.800	25.0	25.0
2,4-Dinitrophenol	.050	.010		
4-Nitrophenol	.050	.010		
Dibenzofuran		.800		25.0
2,4-Dinitrotoluene		.200		25.0
2,6-Dinitrotoluene		.200		25.0
Diethylphthalate		.010		
4-Chlorophenyl-phenylether		.400		25.0
Fluorene		.900		25.0
4-Nitroaniline		.010		
4,6-Dinitro-2-methylphenol		.010		
N-Nitrosodiphenylamine		.010	25.0	
4-Bromophenyl-phenylether		.100		25.0

Semivolatile Compound	Minimum RRF		Maximum %RSD	
	1988	1990	1988	1990
Hexachlorobenzene		.100		25.0
Pentachlorophenol		.050	25.0	25.0
Phenanthrene		.700		25.0
Anthracene		.700		25.0
Carbazole		.010		
Di-n-butylphthalate		.010		
Fluoranthene		.600	25.0	25.0
Pyrene		.600		25.0
Butylbenzylphthalate		.010		
3,3'-Dichlorobenzidene		.010		
Benzo(a)anthracene		.800		25.0
bis(2-Ethylhexyl)phthalate		.010		
Chrysene		.700		25.0
Di-n-octyl phthalate		.010	25.0	
Benzo(b)fluoranthene		.700		25.0
Benzo(k)fluoranthene		.700		25.0
Benzo(a)pyrene		.700	25.0	25.0
Indeno(1,2,3-cd)pyrene		.500		25.0
Dibenz(a,h)anthracene		.400		25.0
Benzo(g,h,i)perylene		.500		25.0
Nitrobenzene-d <sub>5</sub>		.200		25.0
2-Fluorobiphenyl		.700		25.0
Terphenyl-d <sub>14</sub>		.500		25.0
Phenol-d <sub>5</sub>		.800		25.0
2-Fluorophenol		.600		25.0
2,4,6-Tribromophenol		.010		
2-Chlorophenol-d <sub>4</sub>		.800		25.0
1,2-Dichlorobenzene-d <sub>4</sub>		.400		25.0

Note that the 1990 CLP SOW allows 4 compounds with an RRF >.010 requirement and a specified %Diff to be noncompliant. However the RRF's for those four compounds must be greater than or equal to .010, and the %Diff must be less than or equal to 40.0 for the continuing calibration to be acceptable.

## 6.0 ANALYSIS

### 6.1 Analytical Shift

The methodologies require that all quality assurance and sample analyses be done within time limits to maintain a substantive association to calibration and quality assurance operations.

#### 6.1.1 Time Definition

The start of the analytical shift occurs at the injection time of the decafluorotriphenylphosphine (DFTPP) tuning check solution which **passes tuning criteria**. All samples and quality assurance operations must be injected before the shift duration (from the injection of the tuning solution) elapses.

#### 6.1.2 Methodology Specifications

The durations of analytical shifts vary between methodologies. Method 625 designates a shift as a "day", which is a continuous operational run no longer than a 24 hour period. Twelve hours is specified as the analytical shift for 8270 and CLP methodologies, whereas Method 525 uses eight hours as the shift duration.

#### 6.1.3 Shift Quality Assurance Requirements

Before samples are analyzed in a shift, the following quality assurance criteria must be passed: a DFTPP tune and an initial or continuing calibration, in that order. **Prior to the DFTPP tune, inject 1 ul of the column conditioning solution to assist the satisfaction of tune criteria.** A laboratory extraction blank and matrix spikes (for every group of samples in an extraction process) are run as a samples, but not necessarily on every shift. For Method 525, a fortified laboratory blank must be analyzed each shift.

### 6.2 Target Analytes

Semivolatile targets are listed below with practical quantitation limits (PQL's), in **ug/L for water samples and ug/Kg for soils**, achievable by Kiber Analytical procedures at **all times**.

Routinely, significantly lower limits are achieved. The PQL's listed are based upon undiluted dry-weight samples. Diluted samples and samples with a significant amount of moisture result in PQL's which are higher by the factor of dilution and/or dry/wet weight ratio. PQL's achieved by Method 525 are designated with D.

Detection limit studies are performed whenever the analytical system is grossly modified or every six months, whichever is sooner. The reportable detection limits have been determined by the procedure published in 40 CFR 136, Appendix B.

Although pesticides and PCB's can be analyzed by GC/MS, Kiber Analytical uses GC/EC methods when lower quantitation limits are desired or required. Polychlorinated dibenzofurans and dibenzo-p-dioxins can be screened by normal GC/MS methods, but must be quantitated by Method 8280 which is not currently performed in this laboratory.

Please note that many other semivolatile targets can be analyzed if an acceptable standard is available and Kiber Analytical methodologies can be shown to generate accurate and reproducible results.

Analytes on a target list are noted with the following codes: P=Priority Pollutant, T=Toxic/Target Compound, X=Appendix IX, C=California list, D= Drinking water targets, R=LDRL and L=TCLP. Bold T and -T codes indicate compounds added or deleted, respectively, in the 1990 CLP Statement of Work.

SEMIVOLATILE ANALYTE	DW/WATER/SOIL PQLs	LIST CODES
Acenaphthene	10/330	PTX
Acenaphthylene	1D/10/330	PTX
Acetophenone (1-Phenylethanone)	10/330	X
2-Acetylaminofluorene (2-AAF)	20/670	X
Alachlor (Lasso)	3D	D
Aldrin	1D/10/330	PTXCD
4-Aminobiphenyl	10/330	X
Aniline (Benzeneamine)	10/330	X
Anthracene	1D/10/330	PTX
Aramite	10/330	X
Atrazine	3D	D
Benz(a)anthracene	2D/10/330	PTX

SEMIVOLATILE ANALYTE	WATER/SOIL PQLs	LIST CODES
Benzidine	50/1700	P
Benzo(b)fluoranthene (3,4-Benzofluoranthene)	3D/10/330	PTX
Benzo(k)fluoranthene	3D/10/330	PTX
Benzoic acid	50/1700	-T
1,4-Benzoquinone (Quinone), (p-Benzoquinone)	10/330	
Benzo(ghi)perylene	1D/10/330	PTX
Benza(a)pyrene	1D/10/330	PTX
Benzyl alcohol (Benzenemethanol)	20/670	-TX
alpha-BHC	10/330	PTXC
beta-BHC	10/330	PTXC
gamma-BHC (Lindane)	1D/10/330	PTXCD L
delta-BHC	10/330	PTXC
bis(2-Chloroethoxy)methane	10/330	PTXC
bis(2-Chloroethyl)ether	10/330	PTXC
bis(2-Chloroisopropyl)ether [2,2'-oxybis(1-Chloropropane)]	10/330	PTXC
bis(2-Ethylhexyl)adipate	5D	
bis(2-Ethylhexyl)phthalate	8D/10/330	PTX D
4-Bromophenyl phenyl ether	10/330	PTX
Butylbenzyl phthalate (Benzylbutylphthalate)	5D/10/330	PTX D
Carbazole	10/330	T
Chlordane (total)	10/330	P XCD L
Chlordane (alpha isomer)	1D/20/670	T
Chlordane (gamma isomer)	3D/20/670	T
Chlordane (trans Nonachlor)	1D	
p-Chloroaniline	10/330	TXC
Chlorobenzilate	50/1700	XC
4-Chloro-3-methylphenol (p-Chloro-m-cresol)	20/670	PTXC
2-Chloronaphthalene	10/330	PTXC
2-Chlorophenol	10/330	PTXCD
4-Chlorophenyl phenyl ether	10/330	PTX
3-Chloropropionitrile	10/330	C
Chrysene	3D/10/330	PTX
Cyclohexanone	50/1700	R
4,4'-DDD	10/330	PTXC
4,4'-DDE	10/330	PTXC

SEMIVOLATILE ANALYTE	WATER/SOIL PQLs	LIST CODES
4,4'-DDT	20/670	PTXC
Diallate	10/330	X
Dibenz(a,h)anthracene	1D/10/330	PTX
Dibenzofuran	10/330	TX
Di-n-butyl phthalate	10D/10/330	PTX D
1,2-Dichlorobenzene (o-Dichlorobenzene)	10/330	PTXC R
1,3-Dichlorobenzene (m-Dichlorobenzene)	10/330	PTXC
1,4-Dichlorobenzene (p-Dichlorobenzene)	10/330	PTXC L
3,3'-Dichlorobenzidene	20/670	PTXC
2,4-Dichlorophenol	10/330	PTXC
2,6-Dichlorophenol	10/330	XC
Dieldrin	10/330	PTXCD
Diethyl phthalate	6D/10/330	PTX D
Dimethoate	20/670	X
3,3'-Dimethoxybenzidine	30/1000	
p-(Dimethylamino)azobenzene	10/330	X
7,12-Dimethylbenz(a)anthracene	10/330	X
3,3'-Dimethylbenzidine	30/1000	X
alpha, alpha-Dimethylphenethylamine	20/670	X
2,4-Dimethylphenol (2,4-Xylenol)	10/330	PTX
Dimethyl phthalate	3D/10/330	PTX D
m-Dinitrobenzene (1,3-Dinitrobenzene)	20/670	X
4,6-Dinitro-2-methylphenol(4,6-Dinitro-o-cresol)	50/1700	PTX D
2,4-Dinitrophenol	50/1700	PTX
2,4-Dinitrotoluene	10/330	PTX D L
2,6-Dinitrotoluene	10/330	PTX
Dinoseb (2-sec-Butyl-4,6-dinitrophenol), (DNBP)	20/670	X D
Di-n-octylphthalate	10/330	PTX D
Diphenylamine	10/330	X
1,2-Diphenylhydrazine (as azobenzene)	10/330	P
Disulfoton	20/670	X
Endosulfan I (alpha-Endosulfan)	10/330	PTXC
Endosulfan II (beta-Endosulfan)	20/670	PTXC
Endosulfan sulfate	10/330	PTX
Endrin	10D/10/330	PTXCD L
Endrin aldehyde	50/1700	PTXC

SEMIVOLATILE ANALYTE	WATER/SOIL PQLs	LIST CODES
Endrin ketone	20/670	T
Ethyl methanesulfonate	10/330	X
Famphur	20/670	X
Fluoranthene	10/330	PTX
Fluorene	1D/10/330	PTX
Heptachlor	2D/20/670	PTXCD L
Heptachlor epoxide (from hydroxide)	3D/10/330	PTXC L
Hexachlorobenzene	2D/10/330	PTXC L
Hexachlorobutadiene	10/330	PTXC L
Hexachlorocyclopentadiene	1D/10/330	PTXCD
Hexachloroethane	10/330	PTXC L
Hexachlorophene	50/1700	XC
Hexachloropropene	10/330	XC
Indeno(1,2,3-cd)pyrene	1D/10/330	PTX
Isodrin	20/670	XC
Isophorone	10/330	PTX D
Isosafrole (total)	10/330	X
Kepone (Chlordecone)	10/330	XC
Methapyrilene	10/330	X
Methoxychlor	3D/20/670	TXCD L
Metolachlor	5D	D
3-Methylcholanthrene	10/330	X
4,4'-Methylenebis(2-chloroaniline)	50/1700	C
Methyl methanesulfonate	10/330	X
1-Methylnaphthalene	10/330	
2-Methylnaphthalene	10/330	TX
Methyl parathion (Parathion methyl)	20/670	X
2-Methylphenol (o-Cresol)	10/330	TX RL
3-Methylphenol (m-Cresol)	10/330	X RL
4-Methylphenol (p-Cresol)	10/330	TX RL
Naphthalene	10/330	PTX
1,4-Napthoquinone	50/1700	X
1-Naphthylamine	50/1700	X
2-Naphthylamine	10/330	X
2-Nitroaniline (o-Nitroaniline)	10/330	TX
3-Nitroaniline (m-Nitroaniline)	30/1000	TX

SEMIVOLATILE ANALYTE	WATER/SOIL PQLs	LIST CODES
4-Nitroaniline (p-Nitroaniline)	40/1300	TX
Nitrobenzene	10/330	PTX RL
2-Nitrophenol	10/330	PTX
4-Nitrophenol	30/1000	PTX
4-Nitroquinoline 1-oxide	50/1700	X
N-Nitrosodi-n-butylamine	10/330	X
N-Nitrosodiethylamine	10/330	X
N-Nitrosodimethylamine	50/1700	P X
N-Nitrosodiphenylamine (as diphenylamine)	10/330	PTX
N-Nitrosodi-n-propylamine (Dipropylnitrosamine)	10/330	PTX
N-Nitrosomethylethylamine	10/330	X
N-Nitrosomorpholine	10/330	X
N-Nitrosopiperidine	10/330	X
N-Nitrosopyrrolidine	10/330	X
5-Nitro-o-toluidine	10/330	X
Parathion	20/670	X
PCB-1016 (Aroclor 1016)	50/1700	PT CD
PCB-1221 (Aroclor 1221)	50/1700	PT CD
PCB-1232 (Aroclor 1232)	50/1700	PT CD
PCB-1242 (Aroclor 1242)	50/1700	PT CD
PCB-1248 (Aroclor 1248)	50/1700	PT CD
PCB-1254 (Aroclor 1254)	75/2500	PT CD
PCB-1260 (Aroclor 1260)	75/2500	PT CD
Pentachlorobenzene	10/330	XC
Pentachloronitrobenzene	20/670	XC
Pentachlorophenol	30D/30/1000	PTXCD L
Phenacetin	30/1000	X
Phenanthrene	2D/10/330	PTX
Phenol	10/330	PTX D
p-Phenylenediamine (1,4-Benzenediamine)	20/670	X
Phorate	20/670	X
2-Picoline (2-Methylpyridine)	30/1000	X
Polychlorinated biphenyls (PCB's)	2D/10/330	XC
Polychlorinated dibenzo-p-dioxins (PCDD's)	10/330	XC
Polychlorinated dibenzofurans (PCDF's)	10/330	XC
Pronamide	20/670	XC

SEMIVOLATILE ANALYTE	WATER/SOIL PQLs	LIST CODES
Pyrene	1D/10/330	PTX
Safrole	20/670	X
Simazine	2D	D
1,2,4,5-Tetrachlorobenzene	10/330	XC
2,3,7,8-Tetrachlorodibenzo-p-dioxin (Dioxin)	10/330	P XCD
2,3,4,6-Tetrachlorophenol	10/330	XC
Tetraethyl dithiopyrophosphate (Sulfotepp)	20/670	X
Thionazin (Diethyl pyrazinyl phosphorothioate)	20/670	X
o-Toluidine (2-Methylbenzenamine)	20/670	X
Toxaphene (total)	90D/100/3300	PTXCD L
1,2,4-Trichlorobenzene	10/330	PTXCD
2,4,5-Trichlorophenol	20/670	TXC L
2,4,6-Trichlorophenol	10/330	PTXCD L
O,O,O-Triethyl phosphorothioate	20/670	X
1,3,5-Trinitrobenzene (sym-Trinitrobenzene)	50/1700	X
Tris(2,3-dibromopropyl)phosphate (Tris-BP)	50/1700	C

## 6.3 Sample Analysis Operations

### 6.3.1 Documentation

All instrumental and data system parameters unique to analytical runs must be documented in the SVO Logbook. The logs are bound and contain information on all runs, successful or not.

In addition, all quality assurance results are generated in a hard copy form after analysis and placed in the SVO Quality Assurance Notebook which is maintained for each instrument.

### 6.3.2 Data System Operations

#### 6.3.2.1 Batch analysis arrangement

Groups of analytical runs are set up in a sequence file using the BEDIT program. Sequence file namrs use the format SEQ\_XX and can be reused. Run files namrs are named with five-digits, consecutively ordered, from 50000 prescribed with ">". Follow procedures outlined in paragraphs 3.2.1 and 3.2.2 of the overall GC/MS protocol to edit BEDIT and generate a BLIST output.

#### 6.3.2.2 Batch analysis

Sequence files containing batch runs are controlled by the BAMON program (paragraph 3.2.3 of GC/MS overview) which is the primary means of starting and stopping analyses.

### 6.3.3 Analytical Operations

#### 6.3.3.1 Integrated instrument operations

The automatic sampler/injector, gas chromatograph, mass spectrometer and data system are configured so that sample injection, instrumental operations and data system acquisition can commence only when all components are at their proper setpoints and conditions.

#### 6.3.3.2 GC/MS operation

Setpoints for the gas chromatograph and mass spectrometer are set within the METHOD file. This analytical setpoint control file is included in the sequence file setup using program BEDIT. A hard copy of the method can be obtained by invoking the LMETH program and should be saved for inclusion with sample paperwork. All GC/MS instrumental operations are controlled by BAMON (paragraph 3.2.3 in the GC/MS Overview).

## 7.0 POST-ACQUISITION DATA PROCESSING

Post-acquisition data processing is performed on data files to validate automatic processing, check for proper chromatographic and spectrometer operation, generate quality assurance, compare quality assurance results to the required criteria, and finalize target analyte identifications and quantitations.

### 7.1 Data File Configuration

#### 7.1.1 Data File Designations

The five-digit file namr is prescribed with characters to designate the type of file. The ">" symbol signifies the raw Data file acquired during GC/MS analysis. A scan directory file is generated automatically at the end of acquisition, is designated by prescript "<", and must be present for file processing. The corresponding Quant Output file is designated by the "^" character. If additional quantitations are performed, use an alternative descriptor "S" for the first and then any unique descriptor such as A or Q (but not V). During CLP forms generation an "=" file is created which contains deliverable contract information. An example of allied files from the same run would be: >52173, <52173 , ^52173, S52173 and =52173.

#### 7.1.2 File Corruption and Modification

Occasionally data file truncation or directory file corruption occurs. Data file abortion occurs when the run is terminated by power disruption, manual request or when the analysis runs out of disc space during acquisition. Directory file corruption occurs when the data file is terminated for the above reasons or the files are accessed at the same time that the directory file is being generated (immediately after acquisition). These events prevent any Quant Output files from being created at that time.

The analyst can regenerate a nonexistent or corrupted directory file from the raw data file by invoking program FIXDA. The data file can then be accessed to determine if the acquisition satisfies all analytical requirements (including run-length long enough to include the last target analyte), or whether it needs to be rerun. Quant Output files are then created through program QT.

## 7.2 Generic Manual Quant Output Validation Procedure

The manual validation process is performed by total ion chromatogram (TIC) examination and processing the target analyte outputs through the QAREA and QDEL programs (paragraphs 3.3.5 and 3.3.7). All target "hits" are checked for confirmation of proper identification, retention time, proper integration and interferences.

### 7.2.1 Total Ion Chromatogram Examination

The total ion chromatogram (TIC) trace produced in the Medium Quant Output format should be appraised. In general, the chromatogram should appear normal with all predicted standard peaks present. Flattened or tailing peaks, poor peak resolution, an aberrant baseline, a lack (or drop-off) of signal, or a shortened scan time could indicate that the analytical system has malfunctioned and operations should be suspended until the cause of the failure is determined and corrected.

A GC column overburden is indicated by gross abundances in the TIC. Indications of overloading are retarded retention times, flattened or rounded peak apices, poor peak resolution in regions of high response and "hump-o-grams". If column overloading occurs, the sample should be reanalyzed at a greater dilution.

The presence of relatively high molecular weight components is exhibited by a response or elevated baseline at the end of the temperature program. Note significant occurrences of this situation as they necessitate extract dilution and possibly cleaning of the gas chromatographic system.

### 7.2.2 Target Analyte Identification Confirmation

Two criteria must be satisfied to verify identification: (1) elution of a sample component at the same relative retention time (RRT) as that of the standard component and (2) correspondence of the sample component and the standard component mass spectrum.

#### 7.2.2.1 Retention time requirements

The sample component RRT must compare within +/- 0.06 RRT units of the RRT of the standard component. For reference, the standard must be run on the same shift as the sample. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.

#### 7.2.2.2 Spectral requirements

For comparison of sample and standard component mass spectra, the reference spectra must have been previously generated from Kiber Analytical's GC/MS instrumentation. The sample spectrum is accessed (by entering "P" as a QAREA command) and compared to the reference spectrum (by then entering "R"). The requirements for qualitative verification by comparison of mass spectra are as follows:

7.2.2.2.1 All ions present in the standard mass spectrum at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) **must** be present in the sample spectrum.

7.2.2.2.2 The relative intensities of ions specified in the above paragraph must agree within plus or minus 20% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be within 30 and 70 percent.

7.2.2.2.3 Ions greater than 10% in the **sample** spectrum but not present in the **standard** spectrum must be considered and accounted for by the analyst making the comparison.

#### 7.2.2.3 Interpreter judgement

Even if a compound identification (ID) cannot be verified by the criteria above, an ID can be validated through the technical judgement of the mass spectral interpretation specialist.

#### 7.2.2.4 Deletion of unconfirmed analytes

The deletion of false positives generated by the Quant process is performed through the QDEL program. When the analyst is finished with the QAREA operations, exit and delete any unconfirmed "hits" by using QDEL.

### 7.2.3 Quantitation Verification

The automated integration of each reported "hit" should be viewed through QAREA, and when not accurate, should be manually corrected following the guidelines in the description of the QAREA program.

The analyst should note any interferences with the quantitation ion and, if possible, remove the contributing interferent without distorting the target integration. If this is not possible, the target should be quantitated with an alternative ion.

### 7.2.3.1 Characteristic quantitation ions

The characteristic quantitation ions of analytes and standards are shown in the following listing. The primary quantitation ions are used (through the ID file) to produce the normal Quant report. If alternative quantitation ions are needed, use one of the secondary ions listed below. Remember, secondary ion quantitations must use the same calibration process (and files) as used for the primary ion operations. In the table below, "D" signifies the primary quantitation ion used in Method 525, if different from other methods

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
Acenaphthene	153	152,154
Acenaphthene-d <sub>10</sub> (internal std.)	164	162,160
Acenaphthylene	152	151,153
Acetophenone (1-Phenylethanone)	105	77,51
2-Acetylaminofluorene (2-AAF)	181	223,180
Alachlor (Lasso)	160	188,237
Aldrin	66	263,220
4-Aminobiphenyl	169	168,170
Aniline (Benzeneamine)	93	66,65
Anthracene	178	179,176
Aramite	191	185,319,334
Atrazine	200	215
Benz(a)anthracene	228	229,226
Benzidine	184	92,185
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzoic acid	122	105,77
1,4-Benzoquinone (Quinone), (p-Benzoquinone)	108	54,82
Benzo(ghi)perylene	276	138,277
Benza(a)pyrene	252	253,125
Benzyl alcohol (Benzenemethanol)	108	79,77
alpha-BHC	183	181,109
beta-BHC	181	183,109
gamma-BHC (Lindane)	183	181,109
delta-BHC	183	181,109
bis(2-Chloroethoxy)methane	93	95,123
bis(2-Chloroethyl)ether	93	63,95
bis(2-Chloroisopropyl)ether [2,2'-oxybis(1-Chloropropane)]	45	77,79

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
bis(2-Ethylhexyl)adipate	129	112,70
bis(2-Ethylhexyl)phthalate	149	167,279
4-Bromophenyl phenyl ether	248	250,141
Butyl benzyl phthalate(Benzyl butyl phthalate)	149	91,206
Carbazole	167	166,139
Chlordane (total)	373	375,377
Chlordane (alpha isomer)	373	375D,377
Chlordane (gamma isomer)	373	375D,377
Chlordane (trans Nonachlor)	409	
p-Chloroaniline	127	129
Chlorobenzilate	139	251,253
4-Chloro-3-methylphenol (p-Chloro-m-cresol)	107	144,142
2-Chloronaphthalene	162	164,127
2-Chlorophenol	128	64,130
2-Chlorophenol-d <sub>4</sub> (surrogate std.)	132	68,134
4-Chlorophenyl phenyl ether	204	206,141
3-Chloropropionitrile	49	54,89
Chrysene	228	226,229
Chrysene-d <sub>12</sub> (internal std.)	240	120,236
Cyclohexanone	98	42,55
4,4'-DDD	235	237,165
4,4'-DDE	246	248,176
4,4'-DDT	235	237,165
Diallate	86	234,128
Dibenz(a,h)anthracene	278	139,279
Dibenzofuran	168	139
Di-n-butyl phthalate	149	150,104
1,2-Dichlorobenzene (o-Dichlorobenzene)	146	111,148
1,2-Dichlorobenzene-d <sub>4</sub> (surrogate std.)	152	115,150
1,3-Dichlorobenzene (m-Dichlorobenzene)	146	111,148
1,4-Dichlorobenzene (p-Dichlorobenzene)	146	111,148
1,4-Dichlorobenzene-d <sub>4</sub> (internal std.)	152	115
3,3'-Dichlorobenzidene	252	254,126
2,4-Dichlorophenol	162	164,98
2,6-Dichlorophenol	162	164,98
Dieldrin	79	263,279
Diethyl phthalate	149	177,150
Dimethoate	87	125,229

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
3,3'-Dimethoxybenzidine	244	201,229
p-(Dimethylamino)azobenzene	120	225,77
7,12-Dimethylbenz(a)anthracene	256	241,257
3,3'-Dimethylbenzidine	212	211,196
alpha, alpha-Dimethylphenethylamine	58	91,42
2,4-Dimethylphenol (2,4-Xylenol)	107	121,122
Dimethyl phthalate	163	194,164
m-Dinitrobenzene (1,3-Dinitrobenzene)	168	122,76
4,6-Dinitro-2-methylphenol	198	182,77
2,4-Dinitrophenol	184	63,154
2,4-Dinitrotoluene	165	63,182
2,6-Dinitrotoluene	165	89,121
Dinoseb (2-sec-Butyl-4,6-dinitrophenol), (DNBP)	211	163,147
Di-n-octylphthalate	149	
Diphenylamine	169	168,167
1,2-Diphenylhydrazine (as azobenzene)	77	105,182
Disulfoton	88	97,125,153
Endosulfan I	195	339,341
Endosulfan II	337	339,341
Endosulfan sulfate	272	387,422
Endrin	263	82,81D
Endrin aldehyde	67	250,345
Endrin ketone	317	67,319
Ethyl methanesulfonate	79	109,97
Famphur	218	125,93
Fluoranthene	202	101,100
Fluorene	166	165,167
2-Fluorobiphenyl (surrogate std.)	172	171
2-Fluorophenol (surrogate std.)	112	64
Heptachlor	100	272,274,160
Heptachlor epoxide	353	355,351,81
Hexachlorobenzene	284	142,249,286
Hexachlorobutadiene	225	223,227
Hexachlorocyclopentadiene	237	235,272
Hexachloroethane	117	201,199
Hexachlorophene	196	198,209,406
Hexachloropropene	213	143,117
Indeno(1,2,3-cd)pyrene	276	138,227

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
Isodrin	66	193,195
Isophorone	82	95,138
Isosafrole (total)	78	334
Kepone	272	274,237,355
Methapyrilene	58	97
Methoxychlor	227	228
Metolachlor	162	238
3-Methylcholanthrene	268	253,267
4,4'-Methylenebis(2-chloroaniline)	231	266,268
Methyl methanesulfonate	80	79,65
1-Methylnaphthalene	142	141
2-Methylnaphthalene	142	141
Methyl parathion (Parathion methyl)	263	109,125
2-Methylphenol (o-Cresol)	108	107,79
3-Methylphenol (m-Cresol)	108	107,79
4-Methylphenol (p-Cresol)	108	107,79
Naphthalene	128	129,127
Naphthalene-d <sub>8</sub> (internal std.)	136	68
1,4-Naphthoquinone	158	104,130
1-Naphthylamine	143	115,116
2-Naphthylamine	143	115,116
2-Nitroaniline (o-Nitroaniline)	65	92,138
3-Nitroaniline (m-Nitroaniline)	138	108,92
4-Nitroaniline (p-Nitroaniline)	138	92,108
Nitrobenzene	77	123,65
Nitrobenzene-d <sub>5</sub> (surrogate std.)	82	128,54
2-Nitrophenol	139	65,109
4-Nitrophenol	109	139,65
4-Nitroquinoline 1-oxide	190	160,116
N-Nitrosodi-n-butylamine	84	57,41
N-Nitrosodiethylamine	102	42,44
N-Nitrosodimethylamine	42	74,44
N-Nitrosodiphenylamine (as diphenylamine)	169	168,167
N-Nitrosodi-n-propylamine (Dipropylnitrosamine)	70	42,101,130
N-Nitrosomethylethylamine	88	42
N-Nitrosomorpholine	116	56,86
N-Nitrosopiperidine	42	114,55
N-Nitrosopyrrolidine	100	69,44

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
5-Nitro-o-toluidine	152	106,77,79
Parathion	109	137,291
PCB-1016 (Aroclor 1016)	222	260,292
PCB-1221 (Aroclor 1221)	190	222,260
PCB-1232 (Aroclor 1232)	190	222,260
PCB-1242 (Aroclor 1242)	222	256,292
PCB-1248 (Aroclor 1248)	292	362,326
PCB-1254 (Aroclor 1254)	292	362,326
PCB-1260 (Aroclor 1260)	360	362,394
Pentachlorobenzene	250	252,248
Pentachloronitrobenzene	295	237,142
Pentachlorophenol	266	264,268
Perylene-d <sub>12</sub> (internal or surrogate std.)	264	260,265
Phenacetin	108	109,179
Phenanthrene	178	179,176
Phenanthrene-d <sub>10</sub> (internal std.)	188	94,80
Phenol	94	65,66
Phenol-d <sub>5</sub> (surrogate std.)	99	42,71
p-Phenylenediamine (1,4-Benzenediamine)	108	107,80
Phorate	75	47,121
2-Picoline (2-Methylpyridine)	93	66,92
Polychlorinated biphenyls (PCB's)		
monochlorobiphenyls	188	152
dichlorobiphenyls	222	152,224
trichlorobiphenyls	256	258,186
tetrachlorobiphenyls	292	220,290
pentachlorobiphenyls	326	254,256
hexachlorobiphenyls	360	362,290
heptachlorobiphenyls	394	396,324
octachlorobiphenyls	430	428,358
nonachlorobiphenyls	464	462,392
decachlorobiphenyl	498	500,428
Polychlorinated dibenzo-p-dioxins		
hexachlorodibenzo-p-dioxins (HCDD's)	390	388,392,327
pentachlorodibenzo-p-dioxins (PCDD's)	356	354,358,293
tetrachlorodibenzo-p-dioxins (TCDD's)	322	320,257
hexachlorodibenzofurans (HCDF's)	374	372,376,311
pentachlorodibenzofurans (PCDF's)	340	338,342,277
tetrachlorodibenzofurans (TCDF's)	306	304,243

SEMIVOLATILE COMPONENT	QUANTITATION IONS	
	PRIMARY	SECONDARY
Pronamide	173	175,145
Pyrene	202	101,100
Safrole	104	162,103
Simazine	201	203,186
Terphenyl-d <sub>14</sub> (surrogate std.)	244	122,212
1,2,4,5-Tetrachlorobenzene	216	214,218
2,3,7,8-Tetrachlorodibenzo-p-dioxin (Dioxin)	322	320,257
2,3,4,6-Tetrachlorophenol	232	230,131
Tetraethyl dithiopyrophosphate (Sulfotepp)	322	294,266
Thionazin (Diethyl pyrazinyl phosphorothioate)	107	248,143
o-Toluidine (2-Methylbenzenamine)	106	107,77
Toxaphene (mixture total)	159	231,233
2,4,6-Tribromophenol (surrogate std.)	330	332,141
1,2,4-Trichlorobenzen	180	182,145
2,4,5-Trichlorophenol	196	198,200
2,4,6-Trichlorophenol	196	198,200
O,O,O-Triethyl phosphorothioate	198	65,121
1,3,5-Trinitrobenzene (sym-Trinitrobenzene)	75	213,73
Tris(2,3-dibromopropyl)phosphate (Tris-BP)	137	119,257

#### 7.2.4 Final Output Generation

After assessing the TIC, confirming identifications (and deleting false positives), and correcting any improper integrations, a final hard copy Quant Output report should be generated.

#### 7.3 Output Processing and Quality Assurance Checks

Data checks of the pre-run quality assurance operations should be processed as soon as the acquisition data is available. Passage or failure of the required criteria must be determined as soon as possible. Sample data should be screened in a timely manner to determine if reanalysis must be performed.

##### 7.3.1 Tune Output Processing

After acquisition of the DFTPP tuning solution analysis, access the total ion chromatogram and obtain the spectrum at the top of the peak. Type TUNER,=DFTPP and check the result to see if it passes the appropriate criteria found in paragraph 5.1.3.

If this fails, try other peak spectra or spectrum averaging to satisfy the criteria. Background subtraction can be performed (required for Method 625) if it does not distort the spectrum.

NOTE: In 1990 CLP, the mass spectrum of DFTPP **must** be acquired in the following manner:

Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan prior to the elution of DFTPP.

Obtain a hard copy of the spectrum and a passing TUNER result, and file it in the GC/MS SVO Quality Assurance Notebook for that instrument. After passing the tune criteria, run either the initial or continuing calibration. If all efforts fail to produce a spectrum which passes tuning criteria, perform manual tuning to correct the failure.

### 7.3.2 Initial Calibration Output Processing

#### 7.3.2.1 Automated identification check

After the acquisition of one initial calibration standard, the checking process can begin. Check the Quant Output reports to confirm that all standards analyzed were identified automatically. If any were missed, manually quantitate those compounds using program QT with the option T and determine the cause of the miss. Then use IEDIT to correct the qualifications so that all levels of the standard are recognized. Fully requantitate the standard run through QT. Apply the generic manual process to all compounds.

#### 7.3.2.2 Calibration criteria satisfaction

Update the Calibration file with Quant Output files using program CBUPD. Obtain a calibration report through program CBRPT with the %RSD option. Determine if the results satisfy the appropriate criteria listed in paragraph 5.2.4. If criteria are met, the analyst can continue to the step in the next paragraph. If particular standard levels(s) caused the failure, the analyst can reanalyze those standards **only** and reprocess the initial calibration as above. If criteria are still not met, suspend operations and determine if the system is out of control or if the standards themselves are suspect. When the problem situation is corrected, a full initial calibration may be attempted.

#### 7.3.2.3 ID file calibration

When a satisfactory initial calibration has been produced and documented, recalibrate the ID file (ID\_BCA) with the average RF's by using program CBCAL.

### 7.3.3 Continuing Calibration Output Processing

#### 7.3.3.1 Automated identification check

After the acquisition of the continuing calibration standard, review the Quant Output reports to confirm that all standards analyzed were identified automatically. If any were missed, manually quantitate those compounds using program QT with the option T and determine the cause of the miss. Then use IEDIT to correct the qualifications so that the standard is recognized. Fully requantitate the standard through QT. Apply the generic manual process to all compounds.

#### 7.3.3.2 Criteria satisfaction

Compare the continuing standard RF's to the average RF's from the initial calibration through program CBCHK, which automatically generates a report. Determine if the results satisfy the appropriate continuing calibration criteria listed in paragraph 5.3.2. If criteria are met, the analyst can continue to the step in the next paragraph. If the criteria were not met, reanalyze the standard once and reprocess as above. If criteria are still not met, suspend operations and determine whether the GC/MS system is out of control or if the standard integrity is suspect. When the problem situation is corrected, a continuing calibration may be attempted.

#### 7.3.3.3 ID file calibration

When a satisfactory continuing calibration has been produced and documented, recalibrate the daily ID file (ID\_BDA) with the continuing standard RF's by using program QCAL. Since this program also sets retention time windows based on the daily standard, the standard identifications must be correct! ID\_BDA is used to quantitate CLP-protocol samples only. All other protocols utilize ID\_BCA for quantitation.

#### 7.3.4 Laboratory Blank Output Processing and Criteria

##### 7.3.4.1 Standards identification check

If any standard (internal or surrogate) is not automatically identified by the ID file, then the ID file should be edited through IEDIT so that every standard is identified. Requantitate the laboratory blank through the corrected ID file using program QT.

##### 7.3.4.2 Standards abundance check

The Quant report should be reviewed to check the internal and surrogate standards for satisfaction of the criteria found in paragraphs 7.3.6 or 7.3.7. If **any** fail the required criteria, immediately enter QAREA and correct the integration. If the failure cannot be corrected, reanalyze immediately. If a problem still exists, correct it before any more runs are acquired. If the failure of surrogate criteria cannot be corrected by reanalysis, the laboratory blank and all associated samples must be reextracted.

##### 7.3.4.3 Blank target values check

Perform the generic manual validation procedure. The target analyte concentrations in the laboratory blank must be below certain level to show the absence of contamination. The required criteria is based on the 1990 CLP Statement of Work (except for the differing criteria for Method 525).

The laboratory blank must contain less than or equal to five times the quantitation limit of the phthalate esters. For all other target compounds, the method blank must contain less than or equal to the quantitation limit of any single analyte. For Method 525, background from method analytes should be below the method detection limit.

If the method blank exceeds the limits for contamination above, the analyst must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. If reanalysis cannot correct the contamination, reextraction of the blank and all associated samples should be performed.

### 7.3.5 Sample Output Processing

#### 7.3.5.1 Standards identification check

If any standard (internal or surrogate) is not automatically identified by the ID file, then the ID file should be edited through IEDIT so that every standard is properly identified. Requantitate the sample through the corrected ID file using program QT.

#### 7.3.5.2 Standards abundance check

The Quant report should be reviewed to check the internal and surrogate standards for satisfaction of the criteria found in paragraphs 7.3.6 or 7.3.7. If **any** fail the required criteria, immediately enter QAREA and correct the integration. If the failure cannot be corrected, reanalyze at the analyst's convenience. If the problem persists for many samples, make a new fortification standard, run a new calibration or start the whole shift from the beginning. If surrogate standard recoveries in any sample(s) fail to satisfy criteria and instrumental reanalysis does not correct the failure, the failing sample(s) must be reextracted.

#### 7.3.5.3 Sample validation

Perform the generic manual verification procedure. If the concentration of any analyte exceeds the initial calibration range, that sample must be diluted and reanalyzed.

#### 7.3.5.4 Blanks and spikes

Other blanks (field, trip, holding and laboratory fortified) along with the matrix spike and matrix spike duplicate should be processed as samples.

#### 7.3.5.5 Tentatively identified compound processing

When requested, perform a search of the data file for the twenty highest non-target peaks that have abundances above 10% of the abundance of the nearest (by retention time) internal standard. These are identified (if possible), semi-quantitated, and referred to as tentatively identified compounds (TIC's). Invoke program FTIC by entering: ,\*FTICB, [Data File Namr]. This generates the raw data needed to process TIC's.

Be cautious when utilizing identifications produced by automatic probability matching to the spectral data base. Proper interpretation considers automatic and manual spectral matching, retention time, probability of occurrence and chemical knowledge to tentatively identify unknown components. Unless the analyst is certain of the correct identification, interpretation should be performed by the Lab Director. Semi-quantitation is automatically performed by FTIC except the final concentration estimates involving soils are computed incorrectly. Perform manual calculation to rectify the error in the correction for percent moisture.

#### 7.3.6 Surrogate Standard Quality Assurance Criteria

Surrogate standards are added to **every** sample, blank and spike. The criteria for acceptance are as follows:

##### 7.3.6.1 Surrogate criteria for Methods 625 and 525

Specific criteria for each surrogate are not listed for these methods. However, the responses should not vary more than 50% over the course of a shift. If the response during an acquisition varies more than this, the GC/MS system is out of control and the analysis should be performed again.

##### 7.3.6.2 Surrogate criteria for 8270 and CLP methodologies

The following table lists the required surrogate spike recovery limits in percent of amount spiked. Note different criteria for water in the 1988(1989) CLP and the 1990 CLP Statements of Work.

Surrogate Compound	8270/1988 Water	1990 Water	Soil/Sediment
Nitrobenzene-d <sub>5</sub>	35-114	35-114	23-120
2-Fluorobiphenyl	43-116	43-116	30-115
Terphenyl-d <sub>14</sub>	33-141	33-141	18-137
Phenol-d <sub>5</sub>	10-94	10-110	24-113
2-Fluorophenol	21-100	10-110	25-121
2,4,6-Tribromophenol	10-123	10-123	19-122
2-Chlorophenol-d <sub>6</sub> (advisory)		33-110	20-130(1990)
1,2-Dichlorobenzene-d <sub>4</sub> (advisor)		16-110	20-130(1990)

#### 7.3.6.3 Diluted surrogate standards

If a sample dilution greater than 5:1 is necessary, the surrogate standards are considered "diluted out" and criteria are not considered or enforced.

#### 7.3.7 Internal Standard Quality Assurance Criteria

Internal standard responses and retention times must be evaluated after the data acquisition.

##### 7.3.7.1 Retention time criteria

If the retention time for any internal standard changes by more than 30 seconds, the chromatographic system must be inspected for malfunctions, and corrections made as required. Note that very large compound responses indicate column overloading and can cause retention time shifting.

##### 7.3.7.2 Response criteria

If the extracted ion current profile area (quantitation ion integration) for any internal standard changes by more than a factor of two (-50% to 200%) from the latest daily calibration standard, the analysis should be rerun. If the problem is not corrected by reanalysis or continues in a series of consecutive analyses, the problem may be due to erratic internal abundance(s) in the daily calibration standard, in which case the shift should be restarted. If this is not the cause, the GC/MS system must be inspected for malfunction, and corrections made as appropriate.

In Method 525, the integrated areas of internal standards are not constant because the volume of extract varies. The area ratios, however, should be reasonably constant. The addition of 10 uL of terphenyl-d<sub>14</sub> (500 ug/mL) to the extract can be used periodically to monitor recoveries, which should be in excess of 70%.

#### 7.3.8 Matrix Spike Quality Assurance Criteria

The matrix spikes and matrix spike duplicates are used to evaluate the matrix effect of a sample upon the analytical methodology. Other than for Method 525, the criteria are only advisory and are used to indicate when sample matrix effects occur.

7.3.8.1 Method 525 fortification sample and blank requirements  
Method 525 uses fortification blanks and fortified samples for matrix evaluations (and to determine if established detection limits can be met). **All** target analytes are spiked **before** extraction and treated as a sample.

Calculate the measured concentration and accuracy of each analyte. For each analyte, the accuracy (expressed as a percentage of the spiked value) should be 70-130%. Some analytes, particularly the polycyclic aromatic hydrocarbons with molecular weights greater than 220, are allowed an accuracy of 35-130%. If acceptable accuracy and detection limits cannot be achieved, the problem must be located and corrected before further samples are analyzed.

7.3.8.2 Method 8270/CLP matrix spike/spike duplicate criteria  
The matrix spike and matrix spike duplicate recovery (%R) target limits for each spike compound are listed in the table below. The targets for the maximum relative percent difference (RPD) between the recoveries of the matrix spike and the matrix spike duplicate are also listed.

Matrix Spike Compound	%R Water	RPD Water	%R Soil	RPD Soil
Phenol	12-110	42	26-90	35
2-Chlorophenol	27-123	40	25-102	50
1,4-Dichlorobenzene	36-97	28	28-104	27
N-Nitrosodi-n-propylamine	41-116	38	41-126	38
1,2,4-Trichlorobenzene	39-98	28	38-107	23
4-Chloro-3-methylphenol	23-97	42	26-103	33
Acenaphthene	46-118	31	31-137	19
4-Nitrophenol	10-80	50	11-114	50
2,4-Dinitrotoluene	24-96	38	28-89	47
Pentachlorophenol	9-103	50	17-109	47
Pyrene	26-127	31	35-142	36

The limits for %R in water for phenol has been historically 12-89%. The upper limit was expanded to 110% in the 1990 CLP due to accumulated data.

## 8.0 SYSTEM MAINTENANCE

### 8.1 Gas Supply Scrubbers

The oxygen/moisture trap in the helium supply line should be replaced at least once per year or when indicated.

### 8.2 Instrumentation

All system maintenance operations are recorded in detail in the Instrument Notebook.

#### 8.2.1 Gas Chromatographic System Maintenance

##### 8.2.1.1 Gas chromatographic column

The GC column should be altered or replaced when chromatographic degradation approaches an unacceptable level. Degradation can be indicated by poorly shaped peaks, grossly tailing peaks, unsatisfactory quality assurance results, or loss of retention. Column "clipping" can be used to alter the column performance and prolong satisfactory usage. It is performed by cutting off 5 to 15 inches of the front of the column with a column cutting tool. The system, of course, should be shut down during this procedure. Column rinsing with solvents is not recommended to regenerate columns.

## 8.2.2 Mass Spectrometer Maintenance

### 8.2.2.1 Ion Source cleaning

A dirty source is indicated by loss of overall sensitivity, difficulty in instrumental tuning and, most indicatively, a loss of high mass sensitivity. The source should be removed, disassembled, cleaned, rinsed carefully, reassembled and installed. The lens stack in the source should be disassembled for cleaning **only** when there is very noticeable discoloration or every two years. The ceramic source parts should be heated to 400° C with every other cleaning and soaked in aqua regia (with subsequent rinsing) once every two years or when current can be carried across a ceramic source part. Nylon gloves are worn during all operations after a rinsing step.

### 8.2.2.2 Filament replacement

A total loss of signal indicates a broken filament. Switch to the alternate filament if available. If both filaments are broken, they should both be replaced. If the source is removed for cleaning and one filament is broken, it should be replaced at that time. Broken filaments should be accumulated until only one spare is left (per instrument) and then sent for repair instead of purchasing new replacement.

### 8.2.2.3 Quadrupole rod cleaning

The quadrupole rods should be cleaned when dark ion burns are noted near on the rods. **Do not disassemble the rod cluster.** Clean the burn marks with a series of organic solvents using a cotton applicator, then soak the dirty end in solvent overnight.

## 8.3 Data System

### 8.3.1 File Maintenance

#### 8.3.1.1 File purging

Unneeded file should be purged to make room for acquisitions. Data files should be reviewed monthly through SHODA after logging on with MANAGER. Any files older than one month which are through processing and have been reported, should be purged.

Quant output files accumulate on the Aquarius default cartridge and should be reviewed every two months and purged when no further utilization is apparent.

Archival operations must be completed before file purging.

#### 8.3.1.2 Cartridge packing

Cartridge packing should be performed after any significant file purging. The first cartridge on the disc's cartridge list (SC) should be packed weekly since system operations create and purge files on this disc invisibly.

#### 8.3.2 Manual File Archiving

Kiber Analytical produces backup archival tapes using the streaming process. These operations should be performed monthly before file purging operations are performed.