

N60201.AR.002507  
NS MAYPORT  
5090.3a

RESTORATION ADVISORY BOARD MEETING AGENDA AND SLIDES 19 OCTOBER 1995  
NS MAYPORT FL  
10/19/1995  
RESTORATION ADVISORY BOARD

**AGENDA**  
**NAVSTA Mayport**  
**Restoration Advisory Board (RAB)**  
**Orientation Meeting**  
**October 19, 1995, 6:30 p.m.**

- ▶ Welcome Cheryl Mitchell
  
- ▶ Overhead Presentation Richard Stevens  
*INTRODUCTION TO DATA VALIDATION*
  
- ▶ Overhead Presentation Cheryl Mitchell  
*NAVY ENVIRONMENTAL LEADERSHIP PROGRAM (NELP)*
  
- ▶ Presentation and Discussion RAB Members  
*RFI Group II Report*
  
- ▶ Alternate RAB Member Discussion RAB Community Members
  
- ▶ Other Topics RAB Members
  - Document Mailout
  - Availability Session

# **Introduction to Data Validation**

**Richard Stephens**  
**ABB Environmental Services, Inc.**



**Naval Station Mayport**  
**Restoration Advisory Board**

**October 19, 1995**

# **Resource Conservation and Recovery Act (RCRA)**

## **-Test Methods for Evaluating Solid Waste, SW-846.**

### **-Examples:**

**1000 - General characteristics**

**6000 - ICP metals**

**7000 - Atomic Absorption metals**

**8000 - Organics**

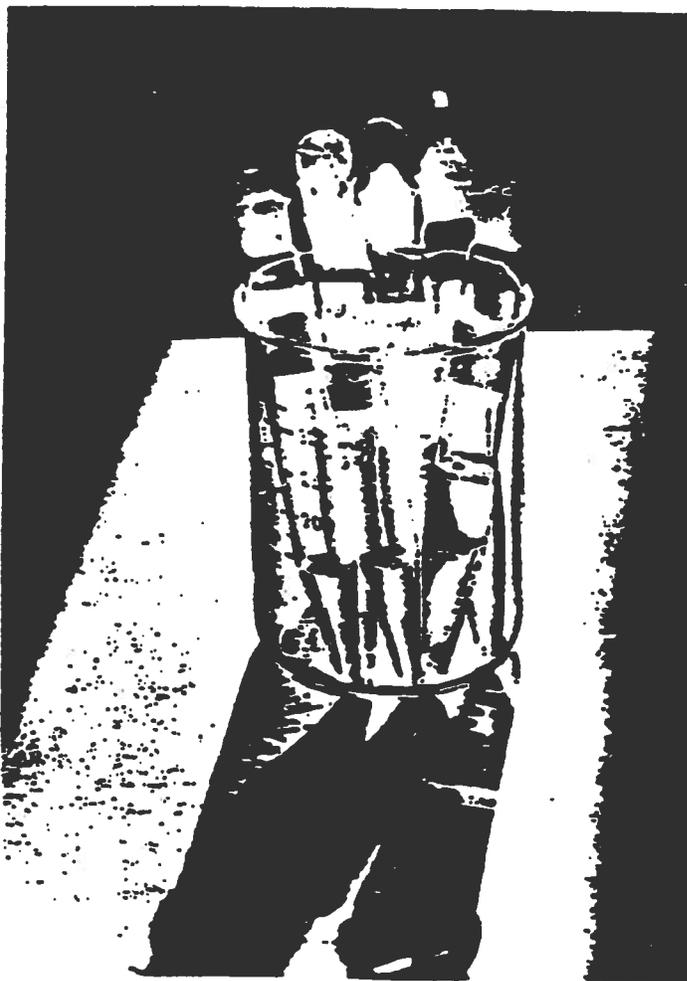
**9000 - Misc. parameters**

Solid Waste



# Test Methods for Evaluating Solid Waste

## Volume IA: Laboratory Manual Physical/Chemical Methods



REPRODUCED BY  
U.S. DEPARTMENT OF COMMERCE  
NATIONAL TECHNICAL  
INFORMATION SERVICE  
SPRINGFIELD, VA 22161

**Comprehensive Environmental Response, Compensation and Liability  
Act (CERCLA)**

**-USEPA Contract Laboratory Program (CLP) Statement of Work  
for Organics Analysis.**

**-USEPA Contract Laboratory Program (CLP) Statement of Work  
for Inorganics Analysis.**

Sample Delivery Group

NAVAL STATION MAYPORT  
SDG SAMPLING ORDER

SDG NO: M3013      MATRIX: SURFACE/BORING SOIL  
LABORATORY: GAL REDDING, CA

SAMPLE #	SAMPLE ID	DATE	TIME	QC ID	TICS	COMMENTS	TRIP BLANK	DATE	TIME
Source	01Y003	05/03/95	09:20			Organic-Free Water System Blank	01T017	05/04/95	09:15
Rinsate	01R015	05/03/95	10:10			Equipment Rinse Blank	01T017	05/04/95	09:15
MS	01S00101MS	05/03/95	11:20	Matrix Spike			01T017	05/04/95	09:15
MSD	01S00101MSD	05/03/95	11:20	Matrix Spike Dup			01T017	05/04/95	09:15
1	01S00101	05/03/95	11:20				01T017	05/04/95	09:15
2	01S00101D	05/03/95	11:20	Duplicate		Field Duplicate	01T017	05/04/95	09:15
3	01B00109	05/03/95	11:50		X		01T017	05/04/95	09:15
4	01S00201	05/03/95	12:20				01T017	05/04/95	09:15
5	01B00208	05/03/95	12:45				01T017	05/04/95	09:15
6	01S00301	05/03/95	14:45				01T017	05/04/95	09:15
7	01S00401	05/03/95	15:00				01T017	05/04/95	09:15
8	01B00308	05/03/95	15:25				01T017	05/04/95	09:15
9	01B00409	05/03/95	16:00				01T017	05/04/95	09:15
10	01S00501	05/03/95	16:40		X		01T017	05/04/95	09:15
11	01B00508	05/03/95	17:10				01T017	05/04/95	09:15
12	01S00601	05/03/95	17:25				01T017	05/04/95	09:15
13	01B00608	05/03/95	17:45				01T017	05/04/95	09:15
14	01B00608D	05/03/95	17:45	Duplicate		Field Duplicate	01T017	05/04/95	09:15
15	01S00701	05/03/95	18:05				01T017	05/04/95	09:15
16	01B00708	05/03/95	18:30				01T017	05/04/95	09:15
17									
18									
19									
20									

TICS = TENTATIVELY IDENTIFIED COMPOUND LIST REQUESTED FOR GC/MS ANALYSIS.

## 7.2 DELIVERABLES

For Level D QC, a CLP data package shall be delivered. This shall include the summary package and the remainder of the package, which includes initial and continuing calibration, matrix spikes, matrix spike duplicates, blanks, duplicates, surrogate recoveries, chromatograms, mass spectra, and absorbance data. For methods which are not defined by CLP, the calibration information, method blanks, blank/spikes, the chromatograms, absorbance, matrix spikes, and matrix spike duplicates shall be reported. The control charts plotted per Sect. 4 associated with the blank/spikes shall be presented with the data.

For Level C QC, the method blanks, blank/spike, surrogates, matrix spikes, matrix spike duplicates, duplicates, and initial and continuing calibration data shall be reported. Table 7.6 lists the required deliverables. The forms referred to in Table 7.6 are from the current CLP for organics and metals/cyanide. The form numbers will be upgraded as new revisions occur in the CLP, which require changes in form content or numbering.

In Level E, the only information to be submitted is the sample data, method blank data, and the control chart from the blank/spike.

The deliverables shall be presented to the NCR. The forms shall be used when reporting any data in the MPR and in submitting the final data package prior to its inclusion in the appendix and summary tables of the final report. The final data deliverables shall be presented to the NCR at least three weeks prior to issuing the draft of the final report.

Publication 9240.1-05  
PB94-963501  
EPA540/R-94/012  
February 1994

**USEPA CONTRACT LABORATORY PROGRAM  
NATIONAL FUNCTIONAL GUIDELINES  
FOR  
ORGANIC DATA REVIEW**

**Office of Emergency and Remedial Response  
U.S. Environmental Protection Agency  
Washington, DC 20460**

**VOLATILE DATA REVIEW**

*\*\*\* Data review guidelines that are unique to data generated through the Low Concentration Water Method are contained within brackets ( [ ] ) and written in italics. \*\*\**

The volatile data requirements to be checked are listed below:

- I. Holding Times (Method Holding Times)
- II. GC/MS Instrument Performance Check
- III. Initial Calibration
- IV. Continuing Calibration
- V. Blanks
- VI. System Monitoring Compounds
- VII. Matrix Spikes/Matrix Spike Duplicates
- VIII. *Laboratory Control Samples*
- IX. Regional Quality Assurance and Quality Control
- X. Internal Standards
- XI. Target Compound Identification
- XII. Compound Quantitation and Reported Contract Required Quantitation Limits (CRQLs)
- XIII. Tentatively Identified Compounds
- XIV. System Performance
- XV. Overall Assessment of Data

## DATA QUALIFIER DEFINITIONS

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

- U** - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J** - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- N** - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
- NJ** - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ** - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R** - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

I. Holding Times

A. **Review Items:** Form I VOA [Form I LCV], EPA Sample Traffic Report and/or chain-of-custody, raw data, and SDG Narrative.

B. **Objective:**

The objective is to ascertain the validity of the analytical results based on the holding time of the sample from the time of collection to the time of analysis.

C. **Criteria:**

Technical requirements for sample holding times have only been established for water matrices.

The technical holding time criteria for water samples are as follows:

For non-aromatic volatile compounds in cooled (@ 4°C) water samples, the maximum holding time is 14 days from sample collection.

Maximum holding times for purgeable aromatic hydrocarbons in cooled (@ 4°C ± 2°C), acid-preserved (with HCl to pH 2 or below) water samples is 14 days from sample collection.

Water samples that have not been maintained at 4°C (± 2°C) and preserved to a pH of 2 or below should be analyzed within 7 days from sample collection. If insufficient ice is used to ship samples, the laboratory may receive samples with no ice left in the cooler. Under these circumstances, the temperature of the samples may exceed 4°C.

**NOTE:** It is further recommended that volatile compounds in properly preserved (4°C ± 2°C) non-aqueous samples be analyzed within 14 days of sample collection.

The method maximum holding times, which differ from the technical maximum holding times, state that water and soil samples are to be analyzed within 10 days from the validated time of sample receipt (VTSR) at the laboratory.

**D. Evaluation:**

Technical holding times are established by comparing the sampling dates on the EPA Sample Traffic Report with dates of analysis on Form I VOA (*Form I LCV*) and the raw data. Information contained in the complete SDG file should also be considered in the determination of holding times. Verify that the analysis dates on the Form Is and the raw data/SDG file are identical. Review the SDG narrative to determine if samples were preserved. If there is no indication in the SDG narrative or the sample records that there was a problem with the samples (e.g., samples not maintained @ 4°C or containing headspace in the samples), then the integrity of samples can be assumed to be good. If it is indicated that there were problems with the samples, then the integrity of the sample may have been compromised and professional judgement should be used to evaluate the effect of the problem on the sample results.

**E. Action:**

1. If technical holding times are exceeded, document in the data review narrative that holding times were exceeded and qualify the sample results as follows (also see Table 1):
  - a. If there is no evidence that the samples were properly preserved and the technical holding times exceeded 7 days, qualify positive results for aromatic compounds with "J" and sample quantitation limits with "UJ". Use professional judgement to determine if and how non-aromatic volatile compounds should also be qualified.
  - b. If the samples were properly preserved but the technical holding times exceeded 14 days, qualify positive results with "J" and sample quantitation limits with "UJ".

Table 1. Qualification of Volatile Analytes Based on Technical Holding Times

MATRIX	PRESERVED	> 7 DAYS	> 14 DAYS
Water	No	All Aromatics*	All Compounds
	Yes	None	All Compounds
Non-aqueous	No/Yes	Professional Judgement	Professional Judgement

- \* Reviewer should use professional judgement to determine if data for additional compounds require qualification.

#### IV. Continuing Calibration

A. **Review Items:** Form VII VOA [*Form VII LCV*], quantitation reports, and chromatograms.

B. **Objective:**

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing acceptable qualitative and quantitative data. Continuing calibration establishes the 12-hour relative response factors on which the quantitations are based and checks satisfactory performance of the instrument on a day-to-day basis.

C. **Criteria:**

1. Continuing calibration standards containing both target compounds and system monitoring compounds are analyzed at the beginning of each 12-hour analysis period following the analysis of the instrument performance check and prior to the analysis of the method blank and samples.
2. The continuing calibration RRF for volatile target compounds and system monitoring compounds must be greater than or equal to 0.05.
3. The percent difference (%D) between the initial calibration  $\overline{RRF}$  and the continuing calibration RRF must be within  $\pm 25.0\%$ .

*[For data generated through the Low Concentration Water Method: The percent difference (%D) between the initial calibration  $\overline{RRF}$  and the continuing calibration RRF must be within  $\pm 30.0\%$ .]*

D. **Evaluation:**

1. Verify that the continuing calibration was run at the required frequency and that the continuing calibration was compared to the correct initial calibration.
2. Evaluate the continuing calibration RRF for all volatile target compounds and system monitoring compounds:
  - a. Check and recalculate the continuing calibration RRF for at least one volatile target compounds associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).
  - b. Verify that all volatile target compounds and system monitoring compounds meet the RRF specifications.

**NOTE:** The criteria employed for data review purposes are different from those defined in the method. The compounds listed in Table 2 (VOA Section III.D.4) have no method maximum %D criteria. The laboratory must meet a minimum RRF criterion of 0.01, however, for data review purposes, the "greater than or equal to 0.05" criterion is applied to all volatile compounds.

3. Evaluate the %D between initial calibration RRF and continuing calibration RRF for one or more compound(s).
  - a. Check and recalculate the %D for one or more volatile target compound(s) associated with each internal standard; verify that the recalculated value(s) agrees with the laboratory reported value(s).
  - b. Verify that the %D is within  $\pm 25.0\%$  for all volatile target compounds and system monitoring compounds. Note those compounds which have a %D outside the  $\pm 25.0\%$  criterion. The method criteria for an acceptable continuing calibration specifies that up to any 2 volatile target compounds may fail to meet minimum RRF or maximum %D as long as they have RRFs that are greater than or equal to 0.010, and %D of less than or equal to 40.0%. For data review purposes, however, all compounds must be considered for qualification when the %D exceeds the  $\pm 25.0\%$  criterion.
4. If errors are detected in the calculations of either the continuing calibration RRF or the %D, perform a more comprehensive recalculation.

**E. Action:**

1. The reviewer should use professional judgement to determine if it is necessary to qualify the data for any volatile target compound. If qualification of data is required, it should be performed using the following guidelines:
  - a. If the %D is outside the  $\pm 25.0\%$  criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify positive results with "J".
  - b. If the %D is outside the  $\pm 25.0\%$  criterion and the continuing calibration RRF is greater than or equal to 0.05, qualify non-detected volatile target compounds with "UJ".
  - c. If the continuing calibration RRF is less than 0.05, qualify positive results that have acceptable mass spectral identifications with "J" or use professional judgement.

- d. If the continuing calibration RRF is less than 0.05, qualify non-detected volatile target compounds as unusable (R).
2. If the laboratory has failed to provide adequate calibration information, the designated representative should contact the laboratory and request the necessary information. If the information is not available, the reviewer must use professional judgement to assess the data.
3. Whenever possible, the potential effects on the data due to calibration criteria exceedance should be noted in the data review narrative.
4. If calibration criteria are grossly exceeded, this should be noted for TPO action.

7.2.7 Tabulate the area response of the characteristic ions (see Table 1) against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured (Section 7.5.2). The RF is calculated as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

$A_x$  = Area of the characteristic ion for the compound being measured.

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

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

$C_x$  = Concentration of the compound being measured.

7.2.8 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are chloromethane, 1,1-dichloroethane, bromoform, 1,1,2,2-tetrachloroethane, and chlorobenzene. The minimum acceptable average RF for these compounds should be 0.300 (0.250 for bromoform). These compounds typically have RFs of 0.4-0.6 and are used to check compound instability and check for degradation caused by contaminated lines or active sites in the system. Examples of these occurrences are:

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

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

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

7.2.9 Using the RFs from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs).

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

where:

RSD = relative standard deviation.

$\bar{x}$  = mean of 5 initial RFs for a compound.

SD = standard deviation of average RFs for a compound.

$$SD = \sqrt{\frac{\sum_{i=1}^N (x_i - \bar{x})^2}{N - 1}}$$

The %RSD for each individual CCC should be less than 30 percent. This criterion must be met in order for the individual calibration to be valid. The CCCs are:

1,1-Dichloroethene,  
Chloroform,  
1,2-Dichloropropane,  
Toluene,  
Ethylbenzene, and  
Vinyl chloride.

### 7.3 Daily GC/MS calibration:

7.3.1 Prior to the analysis of samples, inject or purge 50-ng of the 4-bromofluorobenzene standard. The resultant mass spectra for the BFB must meet all of the criteria given in Table 3 before sample analysis begins. These criteria must be demonstrated each 12-hr shift.

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

7.3.3 **System Performance Check Compounds (SPCCs):** A system performance check must be made each 12 hr. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum response factor for volatile SPCCs is 0.300 (0.250 for Bromoform). Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

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

$$\% \text{ Difference} = \frac{\overline{RF}_I - RF_C}{\overline{RF}_I} \times 100$$

where:

$\overline{RF}_I$  = average response factor from initial calibration.

$RF_C$  = response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 25%, the initial calibration is assumed to be valid. If the criterion is not met (>25% difference), for any one CCC, corrective action MUST be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before quantitative sample analysis begins.

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

#### 7.4 GC/MS analysis:

##### 7.4.1 Water samples:

7.4.1.1 Screening of the sample prior to purge-and-trap analysis will provide guidance on whether sample dilution is necessary and will prevent contamination of the purge-and-trap system. Two screening techniques that can be used are: the headspace sampler (Method 3810) using a gas chromatograph (GC) equipped with a photo ionization detector (PID) in series with an electrolytic conductivity detector (ECD); and extraction of the sample with hexadecane and analysis of the extract on a GC with a FID and/or an ECD (Method 3820).

Table 5

Relative Response Factor Criteria for Initial and Continuing  
Calibration of Volatile Organic Compounds

Volatile Compound	Minimum RRF	Maximum %RSD	Maximum %Diff
Chloromethane	0.010	none	none
Bromomethane	0.100	20.5	±25.0
Vinyl chloride	0.100	20.5	±25.0
Chloroethane	0.010	none	none
Methylene chloride	0.010	none	none
Acetone	0.010	none	none
Carbon disulfide	0.010	none	none
1,1-Dichloroethene	0.100	20.5	±25.0
1,1-Dichloroethane	0.200	20.5	±25.0
1,2-Dichloroethene (total)	0.010	none	none
Chloroform	0.200	20.5	±25.0
1,2-Dichloroethane	0.100	20.5	±25.0
2-Butanone	0.010	none	none
1,1,1-Trichloroethane	0.100	20.5	±25.0
Carbon tetrachloride	0.100	20.5	±25.0
Bromodichloromethane	0.200	20.5	±25.0
1,2-Dichloropropane	0.010	none	none
cis-1,3-Dichloropropene	0.200	20.5	±25.0
Trichloroethene	0.300	20.5	±25.0
Dibromochloromethane	0.100	20.5	±25.0
1,1,2-Trichloroethane	0.100	20.5	±25.0
Benzene	0.500	20.5	±25.0
trans-1,3-Dichloropropene	0.100	20.5	±25.0
Bromoform	0.100	20.5	±25.0
4-Methyl-2-pentanone	0.010	none	none
2-Hexanone	0.010	none	none
Tetrachloroethene	0.200	20.5	±25.0
1,1,2,2-Tetrachloroethane	0.300	20.5	±25.0
Toluene	0.400	20.5	±25.0
Chlorobenzene	0.500	20.5	±25.0
Ethylbenzene	0.100	20.5	±25.0
Styrene	0.300	20.5	±25.0
Xylenes (total)	0.300	20.5	±25.0
<b>SYSTEM MONITORING COMPOUNDS</b>			
Bromofluorobenzene	0.200	20.5	±25.0
Toluene-d <sub>8</sub>	0.010	none	none
1,2-Dichloroethane-d <sub>4</sub>	0.010	none	none

7.3.2 The internal standards selected in Paragraph 5.1 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion, i.e., for 1,4-dichlorobenzene-d<sub>4</sub> use m/z 152 for quantitation.

7.3.3 Analyze 1 uL of each calibration standard (containing internal standards) and tabulate the area of the primary characteristic ion against concentration for each compound (as indicated in Table 1). Figure 1 shows a chromatogram of a calibration standard containing base/neutral and acid analytes. Calculate response factors (RFs) for each compound as follows:

$$RF = (A_x C_{is}) / (A_{is} C_x)$$

where:

A<sub>x</sub> = Area of the characteristic ion for the compound being measured.

A<sub>is</sub> = Area of the characteristic ion for the specific internal standard.

C<sub>x</sub> = Concentration of the compound being measured (ng/uL).

C<sub>is</sub> = Concentration of the specific internal standard (ng/uL).

7.3.4 The average RF should be calculated for each compound. The percent relative standard deviation (%RSD = 100[SD/RF]) should also be calculated for each compound. The %RSD should be less than 30% for each compound. However, the %RSD for each individual Calibration Check Compound (CCC) (see Table 4) must be less than 30%. The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time units. Late-eluting compounds usually have much better agreement.

7.3.5 A system performance check must be performed to ensure that minimum average RFs are met before the calibration curve is used. For semivolatiles, the System Performance Check Compounds (SPCCs) are: N-nitroso-di-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RF for these compounds SPCCs is 0.050. These SPCCs typically have very low RFs (0.1-0.2) and tend to decrease in response as the chromatographic system begins to deteriorate or the standard material begins to deteriorate. They are usually the first to show poor performance. Therefore, they must meet the minimum requirement when the system is calibrated.

TABLE 4. CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-n-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

#### 7.4 Daily GC/MS calibration:

7.4.1 Prior to analysis of samples, the GC/MS tuning standard must be analyzed. A 50-ng injection of DFTPP must result in a mass spectrum for DFTPP which meets the criteria given in Table 3. These criteria must be demonstrated during each 12-hr shift.

7.4.2 A calibration standard(s) at mid-level concentration containing all semivolatile analytes, including all required surrogates, must be performed every 12-hr during analysis. Compare the response factor data from the standards every 12-hr with the average response factor from the initial calibration for a specific instrument as per the SPCC (Paragraph 7.4.3) and CCC (Paragraph 7.4.4) criteria.

7.4.3 System Performance Check Compounds (SPCCs): A system performance check must be made during every 12 hr shift. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. The minimum RF for semivolatile SPCCs is 0.050. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before analysis begins.

7.4.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs listed in Table 4 are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\overline{RF}_I - RF_C}{\overline{RF}_I} \times 100$$

where:

$\overline{RF}_I$  = average response factor from initial calibration.

$RF_C$  = response factor from current verification check standard.

If the percent difference for any compound is greater than 20, the laboratory should consider this a warning limit. If the percent difference for each CCC is less than 30%, the initial calibration is assumed to be valid. If the criterion is not met (>30% difference) for any one CCC, corrective action MUST be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration MUST be generated. This criterion MUST be met before sample analysis begins.

Exhibit D Semivolatiles -- Section 17  
 Tables/Diagrams/Flowcharts

Table 5

Relative Response Factor Criteria for Initial and Continuing  
 Calibration of Semivolatile Target Compounds and Surrogates

Semivolatile Compounds	Minimum RRF	Maximum %RSD	Maximum %Diff
Phenol	0.800	20.5	±25.0
bis(2-Chloroethyl)ether	0.700	20.5	±25.0
2-Chlorophenol	0.800	20.5	±25.0
1,3-Dichlorobenzene	0.600	20.5	±25.0
1,4-Dichlorobenzene	0.500	20.5	±25.0
1,2-Dichlorobenzene	0.400	20.5	±25.0
2-Methylphenol	0.700	20.5	±25.0
2,2'-oxybis(1-Chloropropane)	0.010	none	none
4-Methylphenol	0.600	20.5	±25.0
N-Nitroso-di-n-propylamine	0.500	20.5	±25.0
Hexachloroethane	0.300	20.5	±25.0
Nitrobenzene	0.200	20.5	±25.0
Isophorone	0.400	20.5	±25.0
2-Nitrophenol	0.100	20.5	±25.0
2,4-Dimethylphenol	0.200	20.5	±25.0
bis(2-Chloroethoxy)methane	0.300	20.5	±25.0
2,4-Dichlorophenol	0.200	20.5	±25.0
1,2,4-Trichlorobenzene	0.200	20.5	±25.0
Naphthalene	0.700	20.5	±25.0
4-Chloroaniline	0.010	none	none
Hexachlorobutadiene	0.010	none	none
4-Chloro-3-methylphenol	0.200	20.5	±25.0
2-Methylnaphthalene	0.400	20.5	±25.0
Hexachlorocyclopentadiene	0.010	none	none
2,4,6-Trichlorophenol	0.200	20.5	±25.0
2,4,5-Trichlorophenol	0.200	20.5	±25.0
2-Chloronaphthalene	0.800	20.5	±25.0
2-Nitroaniline	0.010	none	none
Dimethylphthalate	0.010	none	none
Acenaphthylene	0.900	20.5	±25.0
3-Nitroaniline	0.010	none	none
2,6-Dinitrotoluene	0.200	20.5	±25.0
Acenaphthene	0.900	20.5	±25.0
2,4-Dinitrophenol	0.010	none	none
4-Nitrophenol	0.010	none	none
Dibenzofuran	0.800	20.5	±25.0
2,4-Dinitrotoluene	0.200	20.5	±25.0
Diethylphthalate	0.010	none	none
4-Chlorophenyl-phenylether	0.400	20.5	±25.0
Fluorene	0.900	20.5	±25.0
4-Nitroaniline	0.010	none	none
4,6-Dinitro-2-methylphenol	0.010	none	none
N-Nitrosodiphenylamine	0.010	none	none
4-Bromophenyl-phenylether	0.100	20.5	±25.0
Hexachlorobenzene	0.100	20.5	±25.0
Pentachlorophenol	0.050	20.5	±25.0
Phenanthrene	0.700	20.5	±25.0

7A  
VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: CH2M HILL/LMG

Contract:

Lab Code:

Case No.: M7424

SAS No.:

SDG No.: M7424

Instrument ID: HP1

Calibration Date: 07/05/94

Time: 1113

Lab File ID: EVO3601037.D

Init. Calibration Date(s): 06/20/94 06/21/94

Matrix: (soil/water) WATER Level: (low/med) LOW Column: (pack/cap) CAP

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(\*) = 25.0%

COMPOUND	RRF	RRF50	%D
Chloromethane	#1.769	1.132	36.0#
Bromomethane	1.495	1.001	33.0
Vinyl_Chloride	*1.832	1.456	20.5*
Chloroethane	1.180	1.125	4.6
Methylene_Chloride	1.571	1.461	7.0
Acetone	0.331	0.335	-1.3
Carbon_Disulfide	3.628	3.838	-5.8
Trichlorofluoromethane	3.043	3.151	-3.5
1,1-Dichloroethene	*1.469	1.485	-1.1*
1,1-Dichloroethane	#2.962	2.689	9.2#
1,2-Dichloroethene_(total)	1.785	1.693	5.1
Chloroform	*2.845	2.534	10.9*
1,2-Dichloroethane	1.632	1.495	8.4
2-Butanone	0.135	0.104	22.8
1,1,1-Trichloroethane	0.490	0.504	-2.9
Carbon_Tetrachloride	0.461	0.489	-6.0
Vinyl acetate	0.672	0.634	5.6
Bromodichloromethane	0.562	0.576	-2.5
1,2-Dichloropropane	*0.367	0.348	5.4*
cis-1,3-Dichloropropene	0.520	0.529	-1.7
Trichloroethene	0.412	0.413	-0.4
Dibromochloromethane	0.497	0.497	0.0
1,1,2-Trichloroethane	0.322	0.295	8.3
Benzene	0.923	0.932	-1.0
trans-1,3-Dichloropropene	0.425	0.434	-2.2
2-Chloroethyl_vinyl_ether	0.203	0.224	-10.3
Bromoform	#0.393	0.407	-3.6#
2-Hexanone	0.257	0.209	18.7
4-Methyl-2-pentanone	0.169	0.143	15.3
Tetrachloroethene	0.427	0.426	0.3
1,1,2,2-Tetrachloroethane	#0.670	0.615	8.1#
Toluene	*1.181	1.185	-0.3*
Chlorobenzene	#0.963	0.938	2.6#
Ethylbenzene	*0.469	0.478	-1.8*
Styrene	1.004	1.005	0.0
Xylene_(total)	0.632	0.630	0.4
1,3-Dichlorobenzene	0.848	0.858	-1.2

1A  
VOLATILE ORGANICS ANALYSIS DATA SHEET

AA 10/2/94  
EPA SAMPLE NO.

Lab Name: CH2M HILL/LMG

Contract:

16MW001I

Lab Code:

Case No.: M7424

SAS No.:

SDG No.: M7424

Matrix: (soil/water) WATER

Lab Sample ID: M7424001

Sample wt/vol: 5.0 (g/mL) ML

Lab File ID: EVO4701048.D

Level: (low/med) LOW

Date Received: 06/30/94

% Moisture: not dec. \_\_\_\_\_

Data Analyzed: 07/05/94

Column: (pack/cap) CAP

Dilution Factor: 1.0

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) UG/L	Q
74-87-3	Chloromethane	10	U
74-83-9	Bromomethane	10	U
75-01-4	Vinyl Chloride	10	U
75-00-3	Chloroethane	10	U
75-09-2	Methylene Chloride	10	U
67-64-1	Acetone	4	JB
75-15-0	Carbon Disulfide	5	JB
75-69-4	Trichlorofluoromethane	5	U
75-35-4	1,1-Dichloroethene	5	U
75-34-3	1,1-Dichloroethane	5	U
540-59-0	1,2-Dichloroethene (total)	5	U
67-66-3	Chloroform	5	U
107-06-2	1,2-Dichloroethane	5	U
78-83-1	Isobutyl alcohol	5	U
78-93-3	2-Butanone	200	U
71-55-6	1,1,1-Trichloroethane	10	U
56-23-5	Carbon Tetrachloride	5	U
108-05-4	Vinyl acetate	5	U
75-27-4	Bromodichloromethane	10	U
78-87-5	1,2-Dichloropropane	5	U
10061-01-5	cis-1,3-Dichloropropene	5	U
79-01-6	Trichloroethene	5	U
124-48-1	Dibromochloromethane	5	U
79-00-5	1,1,2-Trichloroethane	5	U
71-43-2	Benzene	5	U
10061-02-6	trans-1,3-Dichloropropene	5	U
110-75-8	2-Chloroethyl vinyl ether	10	U
75-25-2	Bromoform	5	U
591-78-6	2-Hexanone	10	U
108-10-1	4-Methyl-2-pentanone	10	U
127-18-4	Tetrachloroethene	5	U
79-34-5	1,1,2,2-Tetrachloroethane	5	U
108-88-3	Toluene	5	U
108-90-7	Chlorobenzene	5	U

7B  
SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: QAL/LRD Contract: R8711  
 Lab Code: \_\_\_\_\_ Case No.: R8711 SAS No.: \_\_\_\_\_ SDG No.: R8711  
 Instrument ID: 4600 Calibration date: 10/04/94 Time: 1152  
 Lab File ID: 94M1BN3231 Init. Calib. Date(s): 09/26/94 09/27/94  
 Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(\*) = 25.0%

COMPOUND	RRF	RRF50	%D
2-Picoline	0.981	1.206	-22.9
Methyl methanesulfonate	0.221	0.333	-50.7
Ethyl Methanesulfonate	0.627	0.735	-17.2
Acetophenone	1.115	1.277	-14.5
N-Nitrosopiperidine	0.385	0.339	12.0
Phenyl-tert-butylamine	1.628	1.882	-15.6
2,6-Dichlorophenol	0.341	0.281	17.6
N-Nitrosodi-n-butylamine	0.292	0.298	-2.1
1,2,4,5-Tetrachlorobenzene	0.480	0.406	15.4
Pentachlorobenzene	0.386	0.337	12.7
1-Naphthylamine	0.949	1.095	-15.4
2-Naphthylamine	1.035	1.161	-12.2
2,3,4,6-Tetrachlorophenol	0.256	0.191	25.4
Phenacetin	0.381	0.431	-13.1
4-Aminobiphenyl	0.813	0.830	-2.1
Pronamide	0.378	0.337	10.8
p-(Dimethylamino)azobenzene	0.503	0.615	-22.3
7,12Dimethylbenz(a)anthracene	0.747	0.738	1.2
3-Methylcholanthrene	0.815	0.820	-0.6
Pyridine	0.970	1.120	-15.5
N-Nitrosomethylethylamine	0.503	0.604	-20.1
N-Nitrosodiethylamine	0.481	0.557	-15.8
N-Nitrosopyrrolidine	0.554	0.666	-20.2
N-Nitrosomorpholine	0.636	0.690	-8.5
o-Toluidine	1.335	1.530	-14.6
Hexachloropropene	0.171	0.144	15.8
Safrole	0.348	0.310	10.9
Isosafrole	0.644	0.638	0.9
1,4-Napthoquinone	0.174	0.152	12.6
1,3-Dinitrobenzene	0.204	0.196	3.9
5-Nitro-o-toluidine	0.409	0.384	6.1
1,3,5-Trinitrobenzene	0.275	0.190	30.9
4-Nitroquinoline-1-oxide	0.139	0.052	62.6
Methapyrilene	0.553	0.808	-46.1
Aramite	0.112	0.121	-8.0
3,3'-Dimethylbenzidine	0.900	0.965	-7.2
Hexachlorophene	0.240	0.030	87.5
2-Acetylaminofluorene	0.647	0.601	7.1
Pentachloronitrobenzene	0.073	0.059	19.2

1D  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

LWR026

Lab Name: QAL/LRD

Contract: R8711

Lab Code: \_\_\_\_\_ Case No.: R8711

SAS No.: \_\_\_\_\_

SDG No.: R8711

Matrix: (soil/water) WATER

Lab Sample ID: R8711002

Sample wt/vol: 1000 (g/mL) ML

Lab File ID: 94M1BN3235

Level: (low/med) LOW

Date Received: 09/10/94

% Moisture: not dec. \_\_\_\_\_ dec. \_\_\_\_\_

Date Extracted: 09/12/94

Extraction: (SepF/Cont/Sonc) CONT

Date Analyzed: 10/04/94

GPC Cleanup: (Y/N) N pH: \_\_\_\_\_

Dilution Factor: 1.0

CONCENTRATION UNITS:  
(ug/L or ug/Kg) UG/L Q

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) <u>UG/L</u>	Q
95-53-4	o-Toluidine	10	U
100-75-4	N-Nitrosopiperidine	10	U
65-85-0	Benzoic acid	50	U
122-09-8	Phenyl-tert-butylamine	50	U
87-65-0	2,6-Dichlorophenol	10	U
1888-71-7	Hexachloropropene	50	U
924-16-3	N-Nitroso-di-n-butylamine	10	U
106-50-3	p-Phenylenediamine	500	U
94-59-7	Safrole	50	U
95-94-3	1,2,4,5-Tetrachlorobenzene	50	U
120-58-1	Isosafrole	50	U
130-15-4	1,4-Napthoquinone	1000	U
99-65-0	1,3-Dinitrobenzene	10	U
608-93-5	Pentachlorobenzene	50	U
134-32-7	1-Naphthylamine	50	U
58-90-2	2,3,4,6-Tetrachlorophenol	10	U UJ VI.3
91-59-8	2-Naphthylamine	50	U
99-55-8	5-Nitro-o-toluidine	10	U
99-35-4	1,3,5-Trinitrobenzene	10	U UJ VI.3
62-44-2	Phenacetin	10	U
92-67-1	4-Aminobiphenyl	50	U
23950-58-5	Pronamide	10	U
82-68-8	Pentachloronitrobenzene	50	U
56-57-5	4-Nitroquinoline-1-oxide	500	U UJ VI.3
91-80-5	Methapyrilene	50	U UJ VI.3
140-57-8	Aramite	50	U
60-11-7	p-Dimethylaminoazobenzene	10	U
119-93-7	3,3'-Dimethylbenzidine	10	U
53-96-3	2-Acetylaminofluorene	10	U
57-97-6	7,12-Dimethylbenz(a)anthracen	10	U
70-30-4	Hexachlorophene	500	U R VI.3
56-49-5	3-Methylcholanthrene	10	U
122-66-7	1,2-Diphenylhydrazine	10	U
N/A	3 & 4-Methylphenol (2)	10	U
92-87-5	Benzidine	50	U UJ VI.3

(2) - 3-Methylphenol & 4-Methylphenol coelute and are reported as a total of the individual isomers.

V. Blanks

A. **Review Items:** Form I VOA [*Form I LCV*], Form IV VOA [*Form IV LCV*], chromatograms, and quantitation reports.

B. **Objective:**

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. **Criteria:**

1. No contaminants should be found in the blanks.
2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.
3. The method blank must be analyzed on each GC/MS system used to analyze samples for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).
4. A storage blank must be prepared upon receipt of the first samples from an SDG, and stored with samples until analysis. The storage blank must be analyzed once per SDG.
5. An instrument blank must be analyzed after any sample that has saturated ions from a given compound to check that the blank is free of interference and the system is not contaminated.

D. **Evaluation:**

1. Review the results of all associated blanks on the forms and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.
2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary

(Form IV VOA *(Form IV LCV)*) to identify the samples associated with each method blank.

3. Verify that a storage blank has been analyzed and included with each SDG and that the storage blanks are free of contamination.
4. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is/are reported at high concentration(s).

**E. Action:**

If the appropriate blanks were not analyzed with the frequency described in Criteria 2, 3, and 4, and 5 then the data reviewer should use professional judgement to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (methylene chloride, acetone, and 2-butanone), or 5 times (5x) the amount for other volatile target compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

Specific actions are as follows:

1. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.
2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgement to determine if further elevation of the CRQL is required. For the common volatile laboratory contaminants, the results are qualified by elevating the quantitation limit to the concentration found in the sample when the sample concentration is less than 10 times (10x) the blank concentration.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

**Example 1:** Sample result is greater than the Contract Required Quantitation Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	<u>Rule</u>	
	<u>10x</u>	<u>5x</u>
Blank Result	7	7
CRQL	5	5
Sample Result	60	30
Final Sample Result	60U	30U

In the example for the "10x" rule, sample results less than 70 (or 10 x 7) would be qualified as not detected. In the case of the "5x" rule, sample results less than 35 (or 5 x 7) would be qualified as not detected.

**Example 2:** Sample result is less than the CRQL, and is also less than the 5x or 10x multiple of the blank result.

	<u>Rule</u>		
	<u>10x</u>	<u>5x</u>	
Blank Result	6	6	
CRQL	5	5	
Sample Result		4J	4J
Final Sample Result	5U	5U	

Note that data are not reported as 4U, as this would be reported as a detection limit below the CRQL.