

Standard Operating Procedure
Analytical Method

TITLE: Analysis of Base/Neutral and Acid (BNA) Compounds by GC/MS

DEPARTMENT: Semivolatile Organics

APPLICATION: This method is used to determine the concentration of various BNA compounds in water, solid waste and biological tissue samples. Appendix A contains the compounds that may be determined by this method and the detection limits for each compound in reagent water.

REFERENCES: Test Methods for Evaluating Solid Wastes
SW846 Method 8000B (Revision 2, December 1996)
SW846 Method 8270C (Revision 3, December 1996)

Code of Federal Regulations
USEPA Method 625 40CFR Pt. 136, App. A, Ch. 1 (7-1-88 Ed.)

PROCEDURE SUMMARY:

This method provides the gas chromatographic conditions for the separation of the compounds in the extract for the quantitative analysis by mass spectrometry. A volume of a sample extract is injected into a gas chromatograph (GC) and compounds in the GC effluent are analyzed by mass spectrometry (MS).

REVIEWED BY: Glen A. Coder 3/1/02
Glen A. Coder
Acting Semivolatile Supervisor
Date

Julie A. Trivedi 3/1/02
Julie A. Trivedi
Quality Assurance Officer
Date

APPROVED BY: Glen A. Coder 3/1/02
Glen A. Coder
Laboratory Manager
Date

Annual Review

Date:						
Initials:						

SAMPLE EXTRACT HANDLING AND STORAGE

Store all extracts at $4^{\circ} \pm 2^{\circ}$ C in the dark in Teflon-sealed containers until analysis is complete. Sample extracts must be analyzed within 40 days from time of extraction.

INTERFERENCES:

Method interferences may be caused by contaminants (primarily phthalate esters) in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks. Contact with common plastics or rubber products must be avoided.

Matrix interference's may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference's will vary considerably from source to source. The GPC (Gel Permeation Chromatography) cleanup procedure is available for cleaning up the sample extract. The extraction personnel initially determine the need for GPC cleanup. The analyst can request that the sample extracts have GPC cleanup prior to the addition of Internal Standards. This is usually determined by visual inspection of the sample extract or by historical data. Tissue samples should routinely be cleaned by GPC.

APPARATUS AND MATERIALS:

GC/MS:	Hewlett Packard (HP) GC5890 series / MSD5970, MSD5972 or equivalent, capable of scanning 35-500 amu at 1 sec/scan.
GC Autosampler:	HP7673 or equivalent.
Data Processor:	HP ChemStation (acquiring) / HP ChemServer-Target 3 (analysis) or equivalent.
Printer:	HP Laserjet 4 or equivalent
Syringes:	10-1000 μ L Gastight syringes (Hamilton series 1000 or equivalent).
Autosampler Vials:	2 mL with crimp top caps.
GC Column:	Rtx-5MS capillary column, 30 m x 0.32 mm I.D. x 0.5 μ m df with guard column or XTI-5 capillary column, 30 m x 0.25 mm I.D. x 0.25 μ m df with guard column (Restek or equivalent).

Note: The conditions below are typical conditions for this method. The actual conditions used for each instrument will be adjusted to optimize instrument performance.

GC Column Conditions:

Carrier gas - Helium	Injector temperature - 280° C
Flow rate - 1.2 mL/min.	Splitless Injection Flow Rate - 50-60 mL/min.
Linear velocity - 43.1 cm/sec.	Auxillary E pressure control - 50 psi
Detector temp. - 290° C	

Inlet B Pressure Program:

Initial Pressure- 0.2 psi
Initial Time - 0.10 min.
Level 1 Rate - 99 psi/min.
Final 1 Pressure -10 psi
Final 1 Time - 0.40 min.
Level 2 Rate- 99 psi/min.
Final 2 Pressure- 0.2 psi
Final 2 Time - 1.2 min.
Level 3 Rate - 0.3 psi/min.
Final 3 Pressure -10 psi
Final 3 Time - 0.0 min.

GC Temperature Program:

Initial temp. - 40° C
Initial time - 2 min.
Rate 1 - 10° C/min.
Final 1 temp. - 300° C
Final 1 time - 0.0 min.
Rate 2 - 16 C/min.
Final 2 temp. - 320 C
Final 2 time - 7 min.

REAGENTS:

Solvents: Methylene chloride and acetone pesticide grade.

Stock Standards Solutions: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 12 months from the date of preparation.

Calibration Standards: Standard Mixtures containing all compounds including surrogate compounds at 6 concentration levels are prepared from the stock solutions. Each calibration solution is spiked with 40 ng of internal standard solution. One of the concentration levels should be at a concentration near, but above, the method detection limit. This low standard of the calibration curve is the reporting limit known as the estimated quantitation limit (EQL). Shelf-life of the calibration solutions is 6 months from the date of preparation.

Internal Standards: A commercially prepared standard mix at a concentration of 4000 µg/mL is used. This solution is certified by the manufacturer (Restek). Shelf-life of standard solution is 6 months from the date of preparation. See Appendix B.

Surrogate Standards: Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation. See Appendix B.

Matrix Spike/Laboratory
Control Sample Standards:

A commercially prepared stock standard solution is used at a concentration of 100 µg/mL certified by the manufacturer. See Appendix C for the list of compounds in the matrix spike mix. See Appendix G for the list of compounds in the Laboratory Control Sample spike mix.

GC/MS Tuning Standard:

Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or an independent source. Shelf-life of standard solutions is 6 months from the date of preparation. See Appendix B.

GC/MS INITIAL CALIBRATION:

1. GC/MS tuning standard: Inject 1 µL of 50 ng of Decafluorotriphenylphosphine (DFTPP). The average of three scans (the apex and the scan before and after the apex) may be used. Background subtraction is required and must be accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. Compare the mass listing to the tuning criteria in appendix D. The tuning criteria must be met in order to continue calibration or sample analysis.

All subsequent standards, samples, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

The DFTPP tuning standard should include 50 ng each of the following additional compounds, pentachlorophenol, benzidine and DDT in order to assess GC column performance and injection port inertness. The degradation of DDT to DDE and DDD should not exceed 20%. If any observable DDD or DDE peaks are present the % Breakdown must be calculated as shown below and recorded in the injection log.

The responses for benzidine and pentachlorophenol should be normal with no peak tailing. If peak tailing is observed, the peak tailing factor must be calculated as illustrated in Appendix J. The tailing factor for Benzidine must be less than 3.0 and less than 5.0 for pentachlorophenol. The tailing factor must be recorded in the injection log if calculated.

$$\text{\% DDT Breakdown} = \frac{\text{Total peak area of (DDD + DDE)}}{\text{(DDD + DDE + DDT)}} \times 100\%$$

If Breakdown and tailing are not observed, note that DFTPP criteria has been met in the injection log.

If degradation of DDT is excessive and/or the chromatography for benzidine or pentachlorophenol is poor the injection port may require cleaning and replacement of the glass liner, liner insert, and the gold seal. Also 6-12 inches of the column should be cut off. This preventative must be performed prior to sample analysis. If the response for pentachlorophenol continues to be very poor or absent then the column may need to be replaced.

2. Inject 1 μL of each of the calibration standard solutions, SSTD010, SSTD020, SSTD050, SSTD080, SSTD120, and SSTD160. Determine the response factors (RF), the average RF and percent relative standard deviation (%RSD) for each compound.

A.
$$\text{RF} = (A_x C_{is}) / (A_{is} C_x)$$

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

B.
$$\% \text{RSD} = 100[\text{SD} / \text{RF}_{\text{ave}}]$$

The %RSD should be less than or equal to 15% for each compound. The %RSD must not exceed 30% for the Calibration Check Compounds (CCC). (See Appendix D).

Linearity - If the %RSD is less than or equal to 15%, then the average RF is used for calculating the concentration of the compound being measured. If the %RSD exceeds 15%, the analyst must choose the best calibration option for quantitation purposes. Linear regression, quadratic regression, and third order polynomial are the other options used for analyte quantitation. It is not the intent to allow non-linear calibration to be used to compensate for detector saturation at higher concentration or to avoid proper instrument maintenance. Non-linear calibration may not be employed for analytes previously shown to exhibit linear calibration. When the linear model is used, the correlation coefficient must be greater than or equal to 0.99.

South Carolina requires the use of a linear calibration model. Either the %RSD is less than 15% and the average RF is used for quantitation or a linear regression with a correlation coefficient greater than or equal to 0.99 must be used for all analytes listed in SW 846 Method 8270C. Any single point within the calibration curve may be re-run if it appears there was a problem with the injection. Since a calibration curve generally consists of more than 5 calibration standards, some of the responses from the upper end of the curve do not need to be included to reduce the data. At least 5 standards must be used to generate the calibration curve. Dilutions must be performed if the concentration of an analyte exceeds the concentration of the highest calibration standard in the curve used to quantify the sample.

- C. The System Performance Check Compounds (SPCC) must have a minimum RF of 0.050 (See Appendix D).

If these criteria are not met, corrective action is required such as cleaning or replacing the injection port liner and/or capillary column or, recalibration.

If the CCCs are not included in the list of analytes for a project, then all required analytes must meet the 30% RSD criterion.

3. Analysis of Initial Calibration Verification Standard

In order to consider the initial calibration acceptable, an Initial Calibration Verification Standard (ICV) must be analyzed prior to sample analysis. The ICV standard must be from a second source and meet the same criteria as the Continuing Calibration Verification (CCV) standard before the initial calibration may be considered valid.

4. GC/MS Daily Calibration:

- A. GC/MS Tuning Standard (DFTPP). Inject 1 μ L of 50 ng DFTPP and compare the mass listing to the acceptance criteria in Appendix D. The tuning standard must precede each 12-hour analysis sequence.
- B. A midpoint calibration standard (50 ppm) must precede sample analysis. The calibration check response factors are compared to average response factors from the initial calibration.
1. The SPCCs must meet the minimum RF criteria of 0.050.
 2. The CCCs must meet the percent difference (%D) criteria.

$$\%D = \frac{RF_{ave} - RF}{RF_{ave}} \times 100$$

If the percent difference for any compound is greater than 20%, this is considered a warning limit. If the percent difference for each of the CCCs is less than or equal to 20%, then the initial calibration is assumed valid and analysis of samples may proceed. If the 20% criterion is not met for CCCs, corrective action must be taken. Corrective action will consist of re-calibration and instrument maintenance if necessary. If the CCCs are not analytes required for the project, then all required analytes must meet the 20% drift criterion.

3. If the retention time for any internal standard in the continuing calibration check standard changes by more than 30 seconds from that in the mid-point standard level in the most recent calibration sequence, the chromatographic system must be inspected for any malfunction and corrective action must be made.
4. The area counts of the internal standard peaks must be within 50-200% of the area counts obtained in the mid-point standard of the initial calibration curve.

SAMPLE ANALYSIS:

1. All samples, method blanks, laboratory control samples and matrix spikes must be analyzed within 12 hours of a valid DFTPP tuning standard.

2. All samples, method blanks, laboratory control samples, and matrix spike extracts are spiked with 40 ng of internal standard solution mix just prior to analysis.

The internal standard areas of the samples, method blanks, laboratory control samples, and matrix spikes must fall within a factor of two (-50% to +100%) range from the preceding midpoint calibration check standard. In addition, the relative retention times of the internal standards for each sample analysis must fall within a ± 30 second window defined by the midpoint calibration check standard.

3. Surrogate recoveries are calculated using the following equation:

$$\text{Surrogate \% Recovery} = (C_{\text{ex}} / C_{\text{s}}) \times 100$$

Where: C_{ex} = Concentration of analyte in the extract (mg/L).
 C_{s} = Calculated concentration of analyte spiked into extract based on amount spiked (mg/L).

Compare the surrogate recoveries according to the specific matrix to the recovery limits in appendix E. These limits are updated annually.

4. Qualitative sample analysis:
- A. The relative retention time (RRT) for the sample component must compare within ± 0.06 RRT units of the standard component.
- B. The mass spectrum for a sample component should compare to the spectrum of the standard component. Note: These criteria do not overrule the judgment of the analyst.
1. All ions present in the standard mass spectrum greater than 10% should be present in the sample spectrum.
 2. The relative intensities of those ions must agree within $\pm 30\%$ of those ions in the reference spectrum.

5. Quantitative sample analysis:

When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the extracted ion current profile (EICP) of the primary characteristic ion. See Appendix A for primary ion (1°) of each compound. A dilution of the sample shall be performed for any analyte that exceeds the high calibration point of the calibration.

Calculation of the Concentration of the analyte in the extract (C_{ex}).

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

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

RF = Average Response factor from calibration curve.

$$\text{Water: concentration } (\mu\text{g/L}) = \frac{(C_{\text{ex}})(V_{\text{F}})(\text{DF})}{(V_{\text{o}})}$$

$$\text{Soil/Tissue: concentration } (\mu\text{g/kg}) = \frac{(C_{\text{ex}})(V_{\text{F}})(\text{DF})}{(W_{\text{s}})(\text{D})}$$

Where: C_{ex} = Concentration of analyte in the extract (ug/mL)
DF = Dilution factor (if applicable)
 V_{o} = Initial sample volume (L)
 V_{F} = Final extract volume (mL)
 W_{s} = Initial sample weight extracted (kg)
D = % solids (if applicable)

Tissue samples are generally reported on an "as is" basis and are not dry weight corrected.

QUALITY CONTROL:

1. The method blank must meet the surrogate limits (see appendix E). If the blank fails these criteria, all of the associated samples, matrix spikes and laboratory control spikes must be re-extracted.
2. The results of the method blank must be (a.) less than the laboratory's reporting limit (see Appendix A), (b.) less than 5% of the regulatory limit associated with an analyte, or (c.) less than 5% of the sample result for the same analyte, whichever is greater. If the blank contains contamination, the source must be located and eliminated.
3. The sample surrogate recovery acceptance criteria are listed in Appendix E. One acid and one base surrogate are allowed to be outside of the acceptance criteria without performing corrective action. **For samples from the State of South Carolina**, if any sample fails these criteria, the sample must be re-extracted unless it is demonstrated to be a matrix effect.
4. Every batch of samples must contain a Laboratory Control Sample (LCS). The LCS is used to verify method performance in the event of poor recoveries in the Matrix Spike or Matrix Spike Duplicate. The control limits for the LCS should fall within the prescribed limits (see Appendix G).

Since an extensive number of compounds are spiked into the LCS, a small percentage of sporadic marginal failures may be tolerated (i.e., will not trigger re-extraction and analysis of the entire batch). See Appendix I for amount of sporadic failures allowed. The control limits for the LCS must fall within the prescribed limits (see Appendix G). See the METHOD EXCEEDANCES section for exceptions to this tolerance. If more analytes are

outside of the control limits than allowed by the sporadic marginal failures formula, the samples are re-extracted.

Any analytes that are outside of the recovery criteria shall be qualified with a '&'.

5. Sample matrix spike component recoveries should fall within the prescribed limits (see Appendix F). The following guidelines are used to determine whether corrective action is needed for MS/MSD recoveries outside of the control criteria:
 - A. If the laboratory control sample extracted and analyzed with the samples is within the control criteria specified in Appendix G, the sample results should be qualified with a "N" flag and reported. The LCS demonstrates that the analytical system was in control. The MS/MSD failure is attributed to matrix effect. Re-extraction is not necessary. Analytes that exceed the RPD criteria are qualified with an "***".
 - B. If the laboratory control sample extracted and analyzed with the samples is also outside of the control criteria, the samples are re-extracted.
6. Leachate samples (TCLP) have different quality control limits for surrogate percent recoveries, LCS percent recoveries and MS percent recoveries. See Appendix E for the surrogate criteria and Appendix H for the LCS and MS criteria.

METHOD EXCEEDANCES:

1. In the Quality Control Section the following allowance is made related to Internal Standards and Surrogates: If an internal standard or surrogate is outside of the control limits, but no compounds of interest are being quantified by that internal standard, no corrective action is required.
2. The State of South Carolina requires the recoveries for all compounds spiked into the LCS to be within acceptance limits (see Appendix G). For samples from the State of South Carolina: If any analytes are outside of the control limits, the system is out of control and corrective action must be taken.
3. Allowance is made for sporadic marginal exceedances of control limits in LCS/LCSD without further corrective action. Data for the analytes that exceed the control limits shall be qualified. The number of allowances is based on the number of analytes spiked. More information can be found in Appendix I of this SOP.
4. In the Quality Control Section an allowance is made for one acid and one base surrogate to be outside of the acceptance criteria without performing corrective action.

Appendix A
BNA DETECTION LIMITS

<u>COMPOUND</u>	ENCHEM ^a Method Detection Limit (<u>µg/L</u>)	ENCHEM ^b Reporting Limit		1° <u>Ion</u>
		<u>Water</u> (<u>µg/L</u>)	<u>Solid</u> (<u>µg/kg</u>)	
INTERNAL STANDARD: 1,4 DICHLOROBENZENE-D4				
Phenol	0.18	10	330	94
bis(2-Chloroethyl)ether	0.29	10	330	63
2-Chlorophenol	0.32	10	330	128
1,3-Dichlorobenzene	0.31	10	330	146
1,4-Dichlorobenzene	0.41	10	330	146
1,2-Dichlorobenzene	0.38	10	330	146
2-Methylphenol	0.21	10	330	108
2,2'-oxybis(1-Chloropropane)	0.27	10	330	45
4-Methylphenol	0.20	10	330	108
N-Nitrosodi-n-propylamine	0.22	10	330	70
Hexachloroethane	0.55	10	330	117
1,4-Dioxane	3.67	10	330	88
Methyl methacrylate	3.36	10	330	69
Ethyl methacrylate	2.16	10	330	69
2-Picoline	1.77	10	330	93
N-Nitrosomethylethylamine	1.51	10	330	88
Methyl methanesulfonate	1.33	10	330	80
N-Nitrosodiethylamine	1.37	10	330	102
Ethyl methanesulfonate	0.73	10	330	79
N-Nitrosopyrrolidine	2.02	10	330	100
N-Nitrosomorpholine	1.41	10	330	56
3-Methylphenol	0.77	10	330	107
o-Toluidine	1.17	10	330	106
Pentachloroethane	2.70	10	330	117
Pyridine	0.46	10	330	79
N-Nitrodimethylamine	0.51	10	330	42
Aniline	1.68	10	330	93
Benzyl alcohol	0.92	10	330	108
Acetophenone	0.67	10	330	105
INTERNAL STANDARD: NAPHTHALENE-D8				
Nitrobenzene	0.34	10	330	77
Isophorone	0.22	10	330	82
2-Nitrophenol	0.32	10	330	139
2,4-Dimethylphenol	0.30	10	330	107
bis(2-Chloroethoxy)methane	0.24	10	330	93
2,4-Dichlorophenol	0.25	10	330	162
1,2,4-Trichlorobenzene	0.35	10	330	180
Naphthalene	0.27	10	330	128

Appendix A (Continued)
BNA DETECTION LIMITS

<u>Compound</u>	ENCHEM ^a Method	ENCHEM ^b		
	Detection Limit	Reporting Limit		1°
	(<u>µg/L</u>)	<u>Water</u>	<u>Solid</u>	<u>Ion</u>
		(<u>µg/L</u>)	(<u>µg/kg</u>)	
INTERNAL STANDARD: NAPHTHALENE-D8 (Continued)				
4-Chloroaniline	0.38	10	330	127
Hexachlorobutadiene	0.56	10	330	225
4-Chloro-3-methylphenol	0.32	10	330	107
2-Methylnaphthalene	0.26	10	330	142
N-Nitrosopiperidine	1.17	10	330	114
a,a-Dimethylphenethylamine	2.13	20	670	58
2,6-Dichlorophenol	0.78	10	330	162
Hexachloropropene	0.91	20	670	213
N-Nitrosodi-n-butylamine	0.91	10	330	84
o,o,o-Triethylphosphorothioate	2.23	10	330	198
p-Phenylenediamine	18.5	100	3300	108
Safrole	1.37	10	330	162
Isosafrole	1.92	10	330	162
Benzoic acid	4.30	50	1700	122
INTERNAL STANDARD: ACENAPHTHENE-D10				
Hexachlorocyclopentadiene	1.23	10	330	237
2,4,6-Trichlorophenol	0.31	10	330	196
2,4,5-Trichlorophenol	0.41	25	830	196
2-Chloronaphthalene	0.26	10	330	162
2-Nitroaniline	0.23	25	830	65
Dimethylphthalate	0.15	10	330	163
Acenaphthylene	0.22	10	330	152
2,6-Dinitrotoluene	0.20	10	330	165
3-Nitroaniline	0.23	25	830	138
Acenaphthene	0.24	10	330	154
2,4-Dinitrophenol	6.44	25	830	184
4-Nitrophenol	0.92	25	830	109
Dibenzofuran	0.20	10	330	168
2,4-Dinitrotoluene	0.29	10	330	165
Diethylphthalate	0.31	10	330	149
4-Chlorophenyl phenyl ether	0.22	10	330	204
Fluorene	0.24	10	330	166
4-Nitroaniline	0.31	25	830	138
1,2,4,5-Tetrachlorobenzene	0.82	10	330	216
1,4-Naphthoquinone	1.80	10	330	158
1,3-Dinitrobenzene	1.41	10	330	168
Pentachlorobenzene	1.13	10	330	250
1-Naphthylamine	1.44	10	330	143

Appendix A (Continued)
BNA DETECTION LIMITS

<u>Compound</u>	ENCHEM ^a Method Detection Limit (<u>µg/L</u>)	ENCHEM ^b Reporting Limit		
		<u>Water</u> (<u>µg/L</u>)	<u>Solid</u> (<u>µg/kg</u>)	<u>1°</u> <u>Ion</u>
INTERNAL STANDARD: ACENAPHTHENE-D10 (Continued)				
2-Naphthylamine	1.36	10	330	143
2,3,4,6-Tetrachlorophenol	2.28	10	330	232
5-Nitro-o-toluidine	0.96	10	330	152
Diphenylamine	0.79	10	330	169
INTERNAL STANDARD: PHENANTHRENE-D10				
4,6-Dinitro-2-methylphenol	1.75	25	830	198
N-Nitrosodiphenylamine	0.23	10	330	169
4-Bromophenyl phenyl ether	0.23	10	330	248
Hexachlorobenzene	0.31	10	330	284
Pentachlorophenol	3.13	25	830	266
Phenanthrene	0.25	10	330	178
Anthracene	0.17	10	330	178
Carbazole	0.25	10	330	167
Di-n-butylphthalate	0.35	10	330	149
Fluoranthene	0.22	10	330	202
1,3,5-Trinitrobenzene	0.96	10	330	213
4-Aminobiphenyl	0.68	10	330	169
Pentachloronitrobenzene	0.74	10	330	237
4-Nitroquinoline-1-oxide	2.61	10	330	190
Dinoseb	1.35	10	330	211
Methapyrilene	15.9	10	330	97
Benzidine	1.47	50	1700	184
Diallate	2.91	10	330	86
Phenacetine	1.43	10	330	108
Pronamide	1.59	10	330	173
1,2-Diphenylhydrazine	1.03	10	330	77
INTERNAL STANDARD: CHRYSENE-D12				
Pyrene	0.23	10	330	202
Butylbenzylphthalate	0.32	10	330	149
3,3'-Dichlorobenzidine	0.44	10	330	252
Benzo(a)anthracene	0.20	10	330	228
Chrysene	0.19	10	330	228
bis(2-Ethylhexyl)phthalate	0.83	10	330	149
p-Dimethylamino azobenzene	1.02	10	330	225
Chlorobenzilate	0.75	10	330	251

Appendix A (Continued)
BNA DETECTION LIMITS

<u>Compound</u>	ENCHEM ^a Method Detection Limit (<u>µg/L</u>)	ENCHEM ^b Reporting Limit		1° <u>lon</u>
		<u>Water</u> (<u>µg/L</u>)	<u>Solid</u> (<u>µg/kg</u>)	
INTERNAL STANDARD: CHRYSENE-D12 (Continued)				
3,3-Dimethylbenzidine	2.65	10	330	212
2-Acetylaminofluorene	0.89	10	330	181
Aramite	4.27	20	670	185
Kepone	23.3	50	1700	272
INTERNAL STANDARD: PERYLENE-D12				
Di-n-octylphthalate	0.39	10	330	149
Benzo(b)fluoranthene	0.19	10	330	252
Benzo(k)fluoranthene	0.30	10	330	252
Benzo(a)pyrene	0.21	10	330	252
Indeno(1,2,3-cd)pyrene	0.33	10	330	276
Dibenzo(a,h)anthracene	0.28	10	330	278
Benzo(g,h,i)perylene	0.40	10	330	276
7,12-Dimethylbenz(a)anthracene	2.32	10	330	256
3-Methylcholanthrene	1.35	10	330	268
Hexachlorophene	38.2	500	17000	196

^a Method Detection Limit determination, USEPA 40CFR Pt.136, App.B, 1988. Method detection limits are updated periodically, the values currently in use may differ slightly from those published.

^b EN CHEM Reporting Limits based on internal Method Detection Limit determinations, USEPA 40CFR Pt.136, App.B, 1988.

Appendix B

Internal Standards

1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

Surrogate Standards

2-Fluorophenol
Phenol-d6
Nitrobenzene-d5
2-Fluorobiphenyl
2,4,6-Tribromophenol
Terphenyl-d14
1,2-Dichlorobenzene-d4
2-Chlorophenol-d4

GC/MS Tuning Standard

Decafluorotriphenylphosphine (DFTPP)

Appendix C

Matrix spike standard

The spike mixture contains the following components at 100 ug/mL:

Phenol
bis(2-Chloroethyl)ether
2-Chlorophenol
1,3-Dichlorobenzene
1,4-Dichlorobenzene
1,2-Dichlorobenzene
2-Methylphenol
2,2-oxybis(1-Chloropropane)
4-Methylphenol
Hexachloroethane
N-Nitroso-di-n-propylamine
Nitrobenzene
Isophorone
2-Nitrophenol
2,4-Dimethylphenol
bis(2-Chloroethoxy)methane
2,4-Dichlorophenol
1,2,4-Trichlorobenzene
Naphthalene
4-Chloroaniline
Hexachlorobutadiene
4-Chloro-3-methylphenol
2-Methylnaphthalene
Hexachlorocyclopentadiene
2,4,6-Trichlorophenol
2,4,5-Trichlorophenol
2-Chloronaphthalene
2-Nitroaniline
Dimethylphthalate
Acenaphthylene
2,6-Dinitrotoluene
3-Nitroaniline

Appendix C (continued)

Matrix spike standard (continued)

Acenaphthene
2,4-Dinitrophenol
Dibenzofuran
4-Nitrophenol
2,4-Dinitrotoluene
Diethylphthalate
Fluorene
4-Chlorophenyl phenyl ether
4-Nitroaniline
4,6-Dinitro-2-methylphenol
N-Nitrosodiphenylamine
4-Bromophenyl phenyl ether
Hexachlorobenzene
Pentachlorophenol
Phenanthrene
Anthracene
Carbazole
di-n-Butylphthalate
Fluoranthene
Pyrene
Butylbenzylphthalate
Benzo(a)anthracene
3,3'-Dichlorobenzidine
Chrysene
bis(2-Ethylhexyl)phthalate
di-n-Octylphthalate
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenzo(a,h)anthracene
Benzo(g,h,i)perylene

Appendix D

GC/MS Tuning Criteria¹
DFTPP

Mass	Ion Abundance Criteria
51	30-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	25-75% of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	> 0.75% of mass 198
441	Present but less than 443
442	40-110% of mass 198
443	15-24% of mass 442

Calibration Check Compounds (CCC)

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

System Performance Check Compounds (SPCC)

N-Nitrosodi-n-propylamine	2,4-Dinitrophenol
Hexachlorocyclopentadiene	4-Nitrophenol

¹ follows CLP-SOW criteria.

² cannot be separated from N-Nitrosodiphenylamine .

Appendix E

SAMPLE ANALYSES
QUALITY CONTROL LIMITS^a

<u>Surrogate Compounds</u>	<u>Water %Rec.</u>	<u>Solids %Rec.</u>	<u>TCLP %Rec.</u>
2-Fluorophenol	(21-79)	(47-98)	(33-72)
Phenol-d5	(18-47)	(48-99)	(19-49)
2-Chlorophenol-d4	(32-99)	(14-95)	(32-99)
1,2-Dichlorobenzene-d4	(54-115)	(33-104)	(64-106)
Nitrobenzene-d5	(57-115)	(43-101)	(63-109)
2-Fluorobiphenyl	(53-131)	(49-106)	(67-109)
2,4,6-Tribromophenol	(29-148)	(34-113)	(56-119)
Terphenyl-d14	(30-151)	(50-114)	(51-123)

^a Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Control limits are based on a mean value \pm 3SD.

Appendix F

SAMPLE MATRIX SPIKE ANALYSES
QUALITY CONTROL LIMITS^a

<u>Matrix Spike Compounds</u>	<u>Water %Rec.</u>	<u>Solids %Rec.</u>
Phenol	(22-60)	(52-104)
bis(2-Chloroethyl)ether	(68-106)	(47-105)
2-Chlorophenol	(72-96)	(48-105)
1,3-Dichlorobenzene	(73-94)	(44-100)
1,4-Dichlorobenzene	(72-96)	(46-100)
1,2-Dichlorobenzene	(72-99)	(46-103)
2-Methylphenol	(65-89)	(50-105)
2,2-oxybis(1-Chloropropane)	(65-104)	(41-114)
4-Methylphenol	(57-85)	(52-105)
Hexachloroethane	(63-106)	(48-102)
N-Nitroso-di-n-propylamine	(71-106)	(52-104)
Nitrobenzene	(66-111)	(51-106)
Isophorone	(67-107)	(42-113)
2-Nitrophenol	(77-100)	(51-104)
2,4-Dimethylphenol	(58-109)	(46-97)
bis(2-Chloroethoxy)methane	(75-102)	(50-109)
2,4-Dichlorophenol	(73-103)	(51-110)
1,2,4-Trichlorobenzene	(70-104)	(48-105)
Naphthalene	(73-104)	(54-103)
4-Chloroaniline	(76-111)	(13-53)
Hexachlorobutadiene	(57-120)	(47-112)
4-Chloro-3-methylphenol	(68-108)	(54-115)
2-Methylnaphthalene	(73-106)	(54-105)
Hexachlorocyclopentadiene	(31-142)	(3-130)
2,4,6-Trichlorophenol	(77-104)	(54-114)
2,4,5-Trichlorophenol	(78-104)	(55-116)
2-Chloronaphthalene	(79-101)	(55-109)
2-Nitroaniline	(67-114)	(53-122)
Dimethylphthalate	(79-103)	(56-117)
Acenaphthylene	(80-103)	(58-111)
2,6-Dinitrotoluene	(76-107)	(57-113)
3-Nitroaniline	(85-108)	(33-73)

Appendix F(Continued)

SAMPLE MATRIX SPIKE ANALYSES
QUALITY CONTROL LIMITS^a

<u>Matrix Spike Compounds</u>	<u>Water %Rec.</u>	<u>Solids %Rec.</u>
Acenaphthene	(81-101)	(56-111)
2,4-Dinitrophenol	(60-116)	(27-93)
Dibenzofuran	(74-104)	(55-113)
4-Nitrophenol	(11-78)	(48-143)
2,4-Dinitrotoluene	(78-102)	(56-113)
Diethylphthalate	(76-106)	(54-117)
Fluorene	(76-115)	(56-119)
4-Chlorophenyl phenyl ether	(71-122)	(55-121)
4-Nitroaniline	(80-141)	(55-125)
4,6-Dinitro-2-methylphenol	(68-116)	(45-107)
N-Nitrosodiphenylamine	(76-119)	(55-126)
4-Bromophenyl phenyl ether	(67-123)	(43-131)
Hexachlorobenzene	(63-125)	(37-137)
Pentachlorophenol	(63-127)	(51-104)
Phenanthrene	(79-107)	(59-117)
Anthracene	(79-108)	(53-114)
Carbazole	(89-158)	(79-149)
di-n-Butylphthalate	(74-113)	(53-122)
Fluoranthene	(78-110)	(56-122)
Pyrene	(79-111)	(61-115)
Butylbenzylphthalate	(75-112)	(55-116)
Benzo(a)anthracene	(70-125)	(58-113)
3,3'-Dichlorobenzidine	(29-172)	(18-94)
Chrysene	(76-116)	(54-132)
bis(2-Ethylhexyl)phthalate	(72-121)	(47-142)
di-n-Octylphthalate	(75-118)	(52-127)
Benzo(b)fluoranthene	(79-110)	(52-121)
Benzo(k)fluoranthene	(68-128)	(41-138)
Benzo(a)pyrene	(76-117)	(52-121)
Indeno(1,2,3-cd)pyrene	(72-127)	(54-135)
Dibenzo(a,h)anthracene	(68-134)	(51-138)
Benzo(g,h,i)perylene	(76-120)	(51-129)

^a Control limits are updated periodically, the values currently in use may differ slightly from those shown above. Matrix Spike limits are based on a mean value \pm 2SD.

na = not available

Appendix G

LABORATORY CONTROL SAMPLE ANALYSES
QUALITY CONTROL LIMITS^a

LCS Compounds*	Water %Rec.	Solids %Rec.
Phenol	(27-56)	(59-109)
bis(2-Chloroethyl)ether	(58-120)	(54-116)
2-Chlorophenol	(65-106)	(68-105)
1,3-Dichlorobenzene	(58-100)	(53-106)
1,4-Dichlorobenzene	(59-102)	(56-104)
1,2-Dichlorobenzene	(58-107)	(54-107)
2-Methylphenol	(60-99)	(55-122)
2,2-oxybis(1-Chloropropane)	(48-137)	(47-129)
4-Methylphenol	(52-93)	(56-122)
Hexachloroethane	(49-106)	(58-108)
N-Nitroso-di-n-propylamine	(53-133)	(56-117)
Nitrobenzene	(56-122)	(58-108)
Isophorone	(57-128)	(71-101)
2-Nitrophenol	(73-107)	(60-109)
2,4-Dimethylphenol	(58-113)	(51-106)
bis(2-Chloroethoxy)methane	(68-111)	(59-111)
2,4-Dichlorophenol	(5-108)	(5-105)
1,2,4-Trichlorobenzene	(60-106)	(57-108)
Naphthalene	(69-105)	(60-107)
4-Chloroaniline	(71-123)	(DL-84)
Hexachlorobutadiene	(41-118)	(56-113)
4-Chloro-3-methylphenol	(62-117)	(64-114)
2-Methylnaphthalene	(62-113)	(57-106)
Hexachlorocyclopentadiene	(12-153)	(6-181)
2,4,6-Trichlorophenol	(74-108)	(67-107)
2,4,5-Trichlorophenol	(74-110)	(67-109)
2-Chloronaphthalene	(72-106)	(65-107)
2-Nitroaniline	(58-127)	(60-118)
Dimethylphthalate	(75-109)	(67-114)
Acenaphthylene	(72-111)	(63-112)
2,6-Dinitrotoluene	(66-124)	(68-115)
3-Nitroaniline	(77-115)	(16-91)
Acenaphthene	(72-109)	(53-126)
2,4-Dinitrophenol	(46-117)	(9-145)
Dibenzofuran	(73-108)	(60-116)
4-Nitrophenol	(17-60)	(34-146)
2,4-Dinitrotoluene	(65-120)	(70-112)
Diethylphthalate	(65-122)	(56-124)
Fluorene	(60-129)	(60-122)
4-Chlorophenyl phenyl ether	(60-130)	(64-116)
4-Nitroaniline	(60-136)	(53-119)
4,6-Dinitro-2-methylphenol	(61-123)	(32-134)

Appendix G (Continued)

LABORATORY CONTROL SAMPLE ANALYSES
QUALITY CONTROL LIMITS^a

LCS Compounds*	Water %Rec.	Solids %Rec.
N-Nitrosodiphenylamine	(69-122)	(69-109)
4-Bromophenyl phenyl ether	(63-124)	(68-108)
Hexachlorobenzene	(61-122)	(67-107)
Pentachlorophenol	(65-120)	(26-144)
Phenanthrene	(73-113)	(63-115)
Anthracene	(76-113)	(66-108)
Carbazole	(56-154)	(48-155)
di-n-Butylphthalate	(65-123)	(67-113)
Fluoranthene	(73-114)	(77-102)
Pyrene	(71-117)	(69-110)
Butylbenzylphthalate	(63-129)	(68-116)
Benzo(a)anthracene	(64-128)	(71-107)
3,3'-Dichlorobenzidine	(31-174)	(13-90)
Chrysene	(64-122)	(64-116)
bis(2-Ethylhexyl)phthalate	(67-127)	(69-115)
di-n-Octylphthalate	(64-139)	(62-131)
Benzo(b)fluoranthene	(71-118)	(60-125)
Benzo(k)fluoranthene	(68-120)	(55-130)
Benzo(a)pyrene	(77-119)	(60-125)
Indeno(1,2,3-cd)pyrene	(68-126)	(64-130)
Dibenzo(a,h)anthracene	(67-129)	(66-131)
Benzo(g,h,i)perylene	(67-126)	(67-121)

* The LCS spiking compound list may vary from above due to project requirements and scope. The QC control limits for other compounds not listed above will have advisory QC control limits until internal limits are developed.

^a Control limits are updated periodically, the values currently in use may differ slightly from those shown above. LCS Spike limits are based on a mean value \pm 3SD.

na = not available

Appendix H

Toxicity Characteristic Leaching Procedure Laboratory Control Spike and Matrix Spike
QUALITY CONTROL LIMITS^a

<u>Compounds</u>	<u>LCS %REC.</u>	<u>MS %REC.</u>
Pyridine	(25-79)	(33-73)
2-Methylphenol	(49-109)	(52-96)
3-&4-Methylphenol	(86-196)	(29-104)
Hexachloroethane	(57-109)	(54-105)
Nitrobenzene	(70-116)	(69-111)
Hexachlorobutadiene	(61-109)	(44-112)
2,4,6-Trichlorophenol	(62-114)	(38-119)
2,4,5-Trichlorophenol	(53-120)	(62-113)
2,4-Dinitrotoluene	(65-108)	(48-134)
Hexachlorobenzene	(50-116)	(37-115)
Pentachlorophenol	(32-145)	(42-124)
Cresol, total	(10-155)	(15-126)

^a Control limits are updated periodically, the values currently in use may differ slightly from those shown above. LCS Spike limits are based on a mean value \pm 3SD and MS spike limits are based on a mean value \pm 2SD.

Appendix I

DETERMINATION OF SPORADIC MARGINAL FAILURES ALLOWED

N ¹	X
5 - 15	1
16 - 30	2
31 - 45	3
46 - 60	4
61 - 75	5
76 - 90	6
91 - 105	7

N = Number of target analytes spiked.

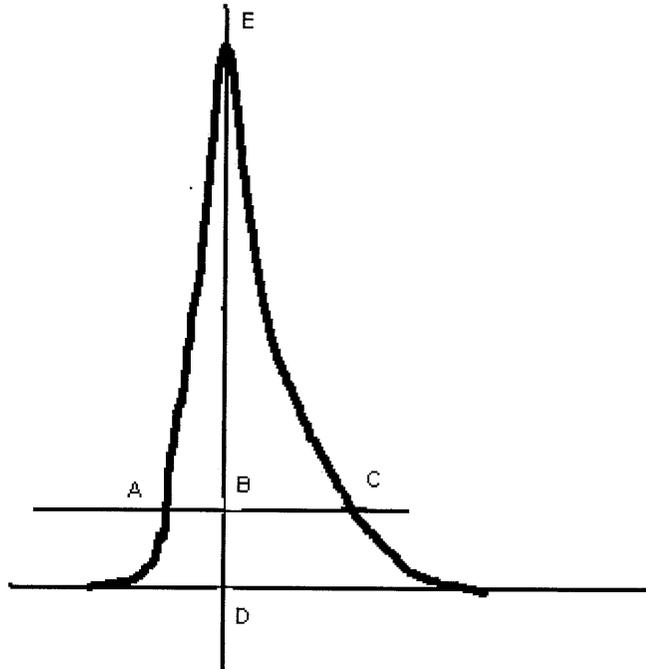
X = Number of Sporadic Marginal Failures (SMF) allowed.

¹ = The number of SMF allowances depend upon the number of target analytes reported from the analysis. For instance, if the full list of target compounds as presented in Appendix A are reported, then five (5) SMFs are allowed. If the Matrix Spike (MS) and/or the Laboratory Control Spike (LCS) includes only a subset of compounds and for surrogates, allow up to one (1) SMF for each B/N and A grouping.

B = Base, N = Neutral and A = Acid compounds.

NOTE: SMFs are used when QC limits have been established. They are not used for compounds with advisory QC limits (i.e., QC limits have not yet been established).

Appendix J
Calculation of Peak Tailing Factor



Sample Calculation

Peak Height = DE = 100mm

10% Peak Height = BD = 10 mm

Peak Width at 10% Peak Height = AC = 23mm

AB = 11 mm

BC = 12 mm

Tailing Factor = BC / AB = 12 / 11 = 1.1