ROUTINE ANALYSIS OF SEMIVOLATILES IN SOIL/SEDIMENT BY GC/MS (EPA/SW-846 Methods 3500B/3541/8000B/8270C) (EPA/SW-846 Methods 3600C/3640A - Optional)

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* These sections affected by Revision 2.0.

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1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) for semi-volatile compounds outlines the preparation and analysis of base/neutral/acid extractable (BNA) compounds in soil/sediment matrices using a gas chromatograph/mass spectrometer (GC/MS). This method is based on Environmental Protection Agency (EPA) Methods SW846/3500B/3541/8000B/8270C and those requirements set forth in the latest approved version of the National Environmental Laboratory Accreditation Committee (NELAC) Quality Systems section. An optional gel permeation chromatography (GPC) cleanup is based on EPA/SW846 Methods 3600C/3640A. A list of target compounds routinely analyzed by the Scientific, Engineering, Response and Analytical (SERAS) Laboratory and the corresponding reporting limits (RLs) are provided in Table 1, Appendix A. This method can be used to quantitate most BNA compounds that are soluble in methylene chloride and capable of being separated on a fused-silica capillary column.

This method may not be changed without the expressed approval of the Organic Group Leader, the Analytical Section Leader and the Quality Assurance Officer (QAO). Only those versions issued through the SERAS document control system may be used. Modifications made to the procedure due to interferences in the samples or for any other reason must be documented in the case narrative and on a nonconformance memo.

2.0 METHOD SUMMARY

Approximately 30 grams (g) of a soil/sediment sample are extracted with 140 mL of 1:1 methylene chloride/acetone mix in a Soxtherm extractor. The extract is concentrated to one milliliter (mL), spiked with an internal standard mixture and subsequently analyzed by GC/MS. Target analytes are identified by comparing the measured mass spectra and retention times with those obtained from calibration standards acquired under the same operating conditions used for the samples. Quantitation of each identified target analyte is calculated based on the internal standard method. Table 2, Appendix A lists the characteristic ions of each target analyte and Table 3, Appendix A lists the internal standards with the corresponding target analytes assigned for quantitation.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

3.1 Sample Storage

Samples should be collected in wide mouth 8-ounce (oz) amber glass containers with Teflon-lined caps. From the time of collection until after analysis, extracts and unused samples must be protected from light and refrigerated at 4 ± 2 degrees Celsius (^{o}C) for the periods specified by SERAS Task Leader (TL) and/or the Work Assignment Manager (WAM) for the project.

Samples and sample extracts must be stored separately from standards in an atmosphere free of all potential contaminants.

3.2 Holding Times

Extraction of soil/sediment samples shall be completed within 7 days from date of collection, and analysis completed within 40 days after sample extraction.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Interferences caused by contaminants in solvents, reagents, glassware and other sample processing hardware, may be introduced during extraction and/or analysis. These interferences may interfere with the identification of target compounds and/or tentatively identified compounds (TICs) (i.e., co eluting peaks) or may cause elevated baselines in the total ion chromatograms. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks on a routine basis. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

Phthalate esters are present in many types of products commonly found in the laboratory. Some plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination may result at any time if good laboratory practices (GLP) or consistent quality control (QC) is not practiced.

5.0 EQUIPMENT/ APPARATUS

The following equipment/apparatus is typically used during the performance of this SOP. Other standard laboratory equipment may be substituted or added, as appropriate.

- Soxtherm extractor, including its accessories (e.g., extraction flask, sample holding vessel, chiller, etc.), manufactured by Gerhardt or equivalent
- Analytical balance, capable of accurately weighing " 0.01 g
- Class "S" weights for calibrating balance
- Spatula, stainless steel or Teflon
- Clear sampling jars, 8-oz amber
- Pyrex glass wool, baked at 400^{O}C for at least 4 hours
- Teflon boiling chips, approximately 10/40 mesh, washed with methylene chloride
- Volumetric flasks, Class A, various volumes ranging from 5 to 500 mL
- Desiccator
- Disposable glass Pasteur pipettes
- TurboVap concentrator, with concentrator cells and racks
- Clean Bath solution, for use in TurboVap II concentrator

- GC autosampler glass vials with crimp caps, 2 mL
- Agilent Technologies 6890 GC and 5972/5973 mass selective detector (MSD) or equivalent, equipped with an autosampler and controlled by the Enviroquant (or equivalent) software
- Restek Rtx-5 MS fused silica capillary column, 30 meter (m) x 0.25 millimeter (mm) inner diameter (ID), 0.5 micron (µm) film thickness (or equivalent)
- Syringes, various microliters (µL) volumes, for spiking and preparation of standards
- Micro syringes, 10 µL and larger, 0.006 inch ID needle
- Vials, 4-mL, for optional GPC cleanup
- Teflon filters, 0.45µm, for filtering extracts for optional GPC cleanup (Gelman Acrodisc CR or equivalent)

6.0 REAGENTS

- Sodium Sulfate, anhydrous powdered reagent grade, heated at 400^{O}C for four hours, cooled in a desiccator, and stored in a glass bottle
- Methylene Chloride, pesticide residue analysis grade or equivalent
- Acetone, GC grade or equivalent
- 1:1 Methylene Chloride/Acetone - Add an equal volume of methylene chloride to an equal volume of acetone.
- Base/Neutral (BN) and Acid Extractable (AE) Stock Surrogate Spiking Solutions, commercially available, typically 100 micrograms per milliliter (µg/mL) for BN and 200 µg/mL for AE in an appropriate solvent mixture. The compounds used are listed below:

Base/Neutrals	Acids
Nitrobenzene-d_5	Phenol-d_5
2-Fluorobiphenyl	2-Fluorophenol
Terphenyl-d_{14}	2,4,6-Tribromophenol

- Alternatively, the above surrogate spiking solution may be prepared from a BN stock spiking solution at 1000 µg/mL and AE stock spiking solution at 2000 µg/mL. Pipet 5.0 mL of each stock spiking solution into a 50 mL Class A volumetric flask and dilute to volume with a water miscible solvent (i.e., acetone, methanol). Surrogate standards are added to all blanks, QC samples, calibration standards and environmental samples.
- Matrix Spike (MS) Stock Solution, commercially available, consisting of the following BN and AE

compounds typically at 100 µg/mL and 200 µg/mL in methanol, respectively. This solution must be a different source from that used for calibration.

Base/Neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

- Laboratory Control Sample (LCS) - With each batch of twenty samples, prepare a LCS at a concentration of 100 and 200 micrograms per liter (µg /L) using the MS stock standard. On a quarterly basis, prepare a LCS from a second source stock standard containing all of the target compounds.

- Internal standard mix, 2000 µg/mL, commercially available, consisting of 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, Perylene-d_{12}. Twenty µL of the internal standard (IS) solution is added to each 1-mL sample extract before analysis, resulting in 40 nanograms/microliter (ng/µL) injection.

- Decafluorotriphenylphosphine (DFTPP), 50 µg/mL, commercially available. The amount of DFTPP in a 1-µL injection is 50 ng.

- Nitrogen, high purity, for evaporation

- Deionized water (DI), organic free reagent water

- Stock GPC Cleanup Standard, consisting of 250 milligrams per milliliter (mg/mL) corn oil, 10 mg/mL bis (2-ethylhexyl)phthalate, 2 mg/mL methoxychlor, 0.2 mg/mL perylene and 0.8 mg/mL of sulfur in methylene chloride (Ultra Scientific CLP-340 or equivalent)

- Working GPC Standard - Dilute 1 mL of stock GPC cleanup standard to 4 mL with methylene chloride

- Stock Calibration Standards, 2000 µg/mL, commercially available in methylene chloride

- Intermediate Calibration Standard, 200 µg/mL in methylene chloride. Add 100 µL of the stock calibration standard to 900 µL of methylene chloride.

- Working Calibration Standards, 10, 20, 50, 80 and 120 µg/mL. Prepare as follows:

Concentration, µg/mL	Volume of 200 µg/mL Intermediate Standard, µL	Volume of Methylene Chloride, µL
10	50	950
20	100	900

50	250	750
80	400	600
120	600	400

NOTE: All of the above-mentioned standard solutions must be stored at -4°C to -10°C (freezer section of the standards refrigerator) in tightly capped vials with Teflon liners. Commercially prepared standard solutions that are received in sealed ampoules may be stored in the shelf section of the standards refrigerator.

NOTE: Premixed certified standards will be stored according to the manufacturer's documented storage requirements. These standards may be kept in storage up to the manufacturer's stated expiration date. Once the standard vials are opened, the standards will be stored with minimal headspace in the freezer for a period not to exceed six months or the manufacturer's expiration date, whichever is less.

NOTE: The IS mixture should be stored in the refrigerator at 4°C. Do not store in the freezer as perylene-d_{12} may fall out of solution.

NOTE: All calibration standards, surrogates, internal standards, and spiking solutions will be prepared and documented in accordance with SERAS SOP #1012, *Preparation of Standard Solutions and Reagents*

7.0 PROCEDURES

7.1 Sample Preparation and Extraction

1. Place the sample container into a fume hood. Open the sample jar and discard any foreign objects such as sticks, leaves, or rocks. Mix the sample thoroughly.

2. Calibrate the balance with class "S" weights immediately prior to weighing any samples. The balance should be calibrated with a weight that is similar to the weight used to extract the sample (i.e., 30 g).

3. Weigh approximately 30 g of each sample to the nearest 0.1g into a 8-oz sampling jar. The sample is then thoroughly mixed with 30-60 g of anhydrous granular sodium sulfate. The sample should have a sandy texture at this point.

4. Prepare a method blank using 30 g of baked sodium sulfate (or sand) at the frequency of one for every 20 samples using the same preparation technique as for the environmental samples.

5. Select a sample to be used for the matrix spike/matrix spike duplicate (MS/MSD). Weigh two additional 30-g portions of this sample to the nearest 0.1 g. A MS/MSD must be prepared for every 10 samples or per project.

   NOTE: The sample to be used for this purpose may be specified on the Chain of Custody (COC)

Record.

6. Cover the bottom of the inner diameter of each sample extraction vessel with a piece of baked glass wool. Place the blended sample and sodium sulfate mixture on the top of the glass wool. Insert the vessel into a Soxtherm extraction flask containing one or two clean boiling chips.

7. Add 0.5 mL of BN and AE stock surrogate spiking solutions to the method blank, LCS, MS/MSD and all environmental samples.

8. Add 0.5 mL of the MS stock solution to the LCS and each MS and MSD.

9. In a fume hood, place 140 mL of 1:1 methylene chloride/acetone into the sample extraction vessel.

10. Connect cooling tubing from the Soxtherm to the chiller. Care must be taken to ensure that cooling is sufficient (e.g., the temperature of the chiller should not be higher than 6^0C.

11. Attach each sample extraction flask to the Soxtherm extractor and extract the sample(s) for 2.5 hours. The typical Soxtherm extraction parameters are listed in Table 4, Appendix A.

12. Allow the extract to cool after the extraction is complete.

13. Fill the TurboVap water bath with approximately one gallon of deionized water mixed with 10-15 drops of Clean Bath solution. Set the water bath temperature at 55^0C.

14. In a fume hood, transfer as much of the combined extracts into 200-mL concentrator tubes. Place the tubes into the TurboVap and begin concentrating by blowing a gentle stream of nitrogen into the tubes so that no solvent splashes out. As the solvent level is reduced, add the remaining extract. Once all of the extract has been added to the concentrator tube and the solvent level is below the 200-mL mark, the flow of nitrogen can be increased to speed up the concentration. Periodically rinse the sides of the tube with methylene chloride.

15. Concentrate the extract until the solvent only remains in the stem of the concentrator tube. This is visible by looking straight down into the cell to see solvent only in the inner circle. At this point, the extract volume is approaching 1 mL and should be monitored carefully. Do not let the extract evaporate to dryness. If gel permeation chromatography (GPC) cleanup is required (refer to Appendix B), concentrate to approximately 4 mL. When removing the cell to add or transfer contents, ensure water bath droplets do not fall into the other extracts.

16. Transfer the extract into a 2-mL GC autosampler vial. The extract is ready for analysis.

7.2 Total Solids

Total solids analysis will be conducted in accordance with SERAS SOP #1843, *Determination of Total Solids in Solid Samples.*

7.3 GC/MS Operating Conditions

The following GC/MS operating conditions are recommended for the Agilent Technologies 6890 GC and 5972/5973 mass selective detector (MSD) system:

Column ID	Restek Rtx-5 MS, 30 m x 0.25 mm ID, 0.5 µm film thickness fused- silica capillary (or equivalent)
Injector Temperature	280^0C
Transfer Temperature	300^0C
Source Temperature	Controlled by transfer line temperature
Temperature Program	50^0C for 0.5 minutes 20^0C /minute (min) to 295^0C, hold for 5 to 8 minutes 25^0C /min to 310^0C, hold for 8 minutes[1]
Injection Mode	1 µL pulse split (with 6:1 split injection)
Mass Range	35 to 450 atomic mass units (amu)[2]
Solvent Delay	3.8 minutes

[1] May be extended to 15 minutes to reduce carryover from samples that contain a high concentration of target or non-target compounds.

[2] May be increased to 500 amu if additional compounds are added to the target compound list.

7.4 Decafluorotriphenylphosphine Tune

The instrument tune must be tuned so that a 50 ng of DFTPP injection produces spectra that will meet the ion abundance criteria listed in Table 4, Appendix A. The tune is acquired using either the apex or the ± one scan. Background subtraction is required and must be accomplished using a single scan no more than 20 scans prior to the elution of the DFTPP. The DFTPP tune criteria must be met every 12 hours during sample analysis. If the software does not indicate what scan was subtracted, the analyst will document the scan number directly on the tune report.

7.5 Initial Calibration

1. Add 20 µL of the internal standard mix to each 1 mL aliquot of the five calibration standards. Do not add the internal standard if using commercially prepared calibration standards that already contain the internal standards.

2. After DFTPP passed the criteria, set up the run using the five-level calibration standards.

3. Calculate and tabulate the relative response factor (RRF) against the concentration for each compound, including the surrogates, by using the equation below. The primary ion from the specific internal standard must be used for quantitation. The average RRF and percent relative standard deviation (%RSD) must also be calculated and tabulated.

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

where:

A_X = Area of the characteristic ion of each target analyte
A_{IS} = Area of the characteristic ion of each internal standard assigned to target analytes
C_{IS} = Concentration of each internal standard, nanograms per microliter (ng/µL)
C_X = Concentration of each target analyte (ng/µL)

4. Use the following equations to calculate and tabulate average RRF and %RSD for all target analytes:

$$RRF_{avg} = \frac{RRF_1 + \ldots + RRF_5}{5}$$

$$\overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

where:

RRF_i = relative response factor for each initial calibration level
n = total number of initial calibration levels

$$SD = \sqrt{\frac{\sum_{n=1}^{5} \left(RF_i - RF_{average}\right)^2}{4}}$$

$$\%RSD = \frac{SD}{RRF_{average}} \times 100$$

where:

N = 5 (number of calibration standards used)
RRFi = individual RRF
RRF_{avg} = average RRF
N = number of calibration standards

The criteria for the average RRF and %RSD for each target analyte are found in Section 9.2.

7.6 Continuing Calibration

1. A check of the initial calibration curve must be performed every 12 hours after an acceptable DFTPP analysis. Sample analysis may begin only after a successful DFTPP tune and a continuing calibration check have been acquired.

2. Inject 1 µL of a 50 µg/mL calibration standard that contains target analytes, surrogates and internal standards. The internal standard concentration is 40 µg/mL.

3. Calculate and tabulate the continuing calibration RRF for each compound.

4. Calculate the percent difference (%D) for the continuing calibration RRF compared to the average RRF from the initial calibration curve.

$$\%D = \frac{RRF_{Daily} - RRF_{Average}}{RRF_{Average}} x\,100$$

The criteria for the continuing RRF and %D are found in Section 9.3.

4. The extracted ion current profile (EICP) area for each internal standard in the continuing calibration must be compared to the internal standard area in the mid-point standard of the current initial calibration. The criterion for comparison is found in section 9.3.

7.7 Sample Analysis

Prior to the analysis of calibration standards, blanks, and/or samples, it is necessary to verify that the GC/MS:

- Met the DFTPP ion abundance criteria listed in Table 4, Appendix A and in Section 9.1. The DFTPP tune criteria must be demonstrated every 12 hours by analyzing 50 ng of DFTPP.

- Successfully passed an initial five-point calibration and/or continuing calibration check. The continuing calibration check must be demonstrated every 12 hours during sample analysis by analyzing a 50 µg/mL BNA standard.

The method blanks, LCS, MS/MSD and samples must be analyzed with the same instrument conditions used for the calibration standards.

1. Add 20 µL of the internal standard mix into the method blank, LCS, MS/MSD, and all sample extracts.

2. Inject 1 µL of the extract for each method blank, LCS, MS/MSD or sample.

3. If a sample extract appears (visual observation) or is known to contain a high concentration of semi-volatile organic compounds, it is advisable to screen the sample along with a 50 µg/mL

calibration standard using gas chromatography/flame ionization detector (GC/FID). Consult the Organic Group Leader when screening the sample and discuss the screening results for subsequent actions to be taken (e.g. an undiluted run may not be necessary due to high concentrations of target or non-target compounds).

4. If the response of any analyte exceeds that of the highest calibration standard, the extract must be diluted so that the analyte response falls within the linear range established in the initial calibration. Ideally, the concentration of the analyte should fall midrange of the curve after dilution.

5. After a dilution is prepared, the internal standard mix is added accordingly, to maintain the required concentration of 40 ng/L of each internal standard in the diluted extract.

7.8 Identification of Target Analytes

The target analytes are identified by comparison of the sample mass spectra with the mass spectra of a calibration standard. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component at the GC relative retention time (RRT) as the standard component
- Correspondence of the sample component and standard component mass spectra

1. For establishing correspondence of the RRT, 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 co-elution 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.

2. For comparison of standard and sample components, reference mass spectra must be obtained from the 50 µg/mL calibration standard. The standard mass spectra may be obtained from the run used to obtain the reference RRTs. In the case of co-elution of standard components, the reference mass spectra from the National Institute of Standard and Technology (NIST) Mass Spectral Library should be used or the analyst can use professional judgment to establish the presence of target analytes. If professional judgment is used, the reason why the analyst chose to use professional judgment must be documented in the case narrative.

3. The requirements for qualitative verification of mass spectra are as follows:

   a. All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion equals 100%) *must* be present in the sample mass spectra

   b. The relative intensities of ions specified in (a) must agree within ±20% between the standard and sample spectra. For example, if an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30-70%.

c. Ions greater than 10% present in the *sample* spectrum but not in the *standard* spectrum must be considered and accounted for by the analyst making the comparison. All target analytes meeting the identification criteria must be reported with their mass spectra. Report the actual value of all target analytes below the quantitation limit with a flag of "J", e.g., "3 J".

4. If a compound cannot be verified by all of the criteria in Step 3 but is identified by the technical judgment of the mass spectral interpretation specialist, the analyst shall report that identification and proceed with the calculation described in Section 8.0. The analyst should report in the case narrative the reasons why the compound is identified.

7.9 Library Search

A library search will be performed for non-target compounds present in the method blank and the samples for the purpose of tentative identification. The 2005 release of the NIST/EPA/NIH Mass Spectral Library (Wiley7n.l) for the "Liberty" instrument or the 1996 release (NBS75k.l) for the "Gordon" instrument containing more than 100,000 spectra will be used.

1. Any non-surrogate organic compounds not listed in Table 1, Appendix A for the combined base/neutral and acid fractions shall be tentatively identified via the NIST mass spectral library. Substances with responses less than 10% of the nearest internal standard are not reported. Only after visual comparison of the sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification.

2. Guidelines for making tentative identification:

- Relative intensities of major ions greater than 10% of the most abundant ion in the reference spectrum should be present in the sample spectrum.
- The relative intensities of the major ions should agree within ±20% between the standard and sample spectra. For example, if an ion has an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30-70%.
- Molecular ions present in reference spectrum should be present in sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
- Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting compounds.

NOTE: Data system library and reduction programs can sometimes create these discrepancies.

3. If all the above conditions for a compound are met and if the Q value of the search is $80%, that

compound will be reported as a tentatively identified compound (TIC). If the Q value is <80% or the mass spectral interpretation specialist indicates that no valid tentative identification can be made, the compound should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, and unknown acid, unknown chlorinated compound). If probable molecular weights can be distinguished, include them on the TIC report. Report only one type of unknown compound per retention time (RT).

4. Up to twenty (20) organic compounds of greatest apparent concentration that are not target analytes shall be identified and reported by a forward library search.

8.0 CALCULATIONS

8.1 Target Compounds

Identified target analytes must be quantitated by the internal standard method. The internal standard used must be the one nearest the retention time to that of the given analyte listed in Table 3, Appendix A. The extracted ion current profile (EICP) area of the characteristic ion of each target analyte listed in Table 2, Appendix A is used for quantitation.

Whether the sample is analyzed after initial calibration or daily continuing calibration, the average relative response factor (RRF_{ave}) is used to calculate the concentrations of identified analytes based on the following equation:

$$Concentration\,(\mu g/kg) = \frac{(A_X)(I_S)(V_T)(DF)}{(A_{is})(RRF_{ave})(W)(S)(V_i)}$$

where:

A_X	=	Area of the characteristic ion of each target analyte
I_S	=	Amount of each internal standard injected (ng)
V_T	=	Volume of the concentrated extract (mL)
DF	=	Dilution factor
A_{IS}	=	Area of the characteristic ion of each internal standard
RRF_{ave}	=	Average relative response factor
W	=	Weight of soil/sediment extracted (kg)
S	=	Decimal percent solid
V_i	=	Injection volume (usually 1 µL)

The following EPA-defined flags will be used in the lab to qualify data:

U: This flag indicates that the compound was analyzed for but not detected

J: This flag indicates an estimated value under the sample RL. Any concentration less than 25% of the RL will not be reported

B: This flag is used when the analyte is found in the associated method blank as well as in the

sample

E: This flag identifies compounds whose concentrations exceed the upper calibration range of the instrument

All target concentrations are reported to three significant figures. For any concentrations reported from diluted runs, be sure to report the corresponding RL. For example, if a compound is run at a 10x dilution to bring the concentration within linear range, the RL must be reported at 100 µg/L instead of 10 µg/L. The RL is based on the lowest standard from the calibration curve multiplied by any dilution factor.

8.2 Tentatively Identified Compounds

An estimated concentration for TICs must be calculated by the internal standard method. The nearest preceding internal standard free of interferences must be used. The equation for calculating the concentration is the same as in Section 8.1, except that area count or peak height of the TICs and their assigned internal standards from the total ion chromatogram is used for calculation. The RRF of both is assumed to be 1.0. All non-target concentrations are reported to one significant figure for concentrations less than 10 and two significant figures for all concentrations greater than or equal to 10.

8.3 Surrogate Spike Recoveries

Calculate surrogate standard recovery on all samples, blanks, and spikes by the following equation:

$$Percent\ Recovery\ (\%R) = \frac{Q_D}{Q_A}\ x\ 100$$

where:

Q_D = Quantity determined by analysis
Q_A = Quantity added to sample

8.4 Matrix Spike Recoveries

Accuracy is calculated from the recovery of the MS/MSDs. Precision is calculated from the relative percent difference (RPD) of the recoveries measured for the MS/MSD pair. Matrix spike recoveries and RPD will be calculated by the following equations:

$$Matrix\ Spike Recovery (\%R) = \frac{SSR - SR}{SA}\ x\ 100$$

where:

SSR = Spike sample result
SR = Sample result

SA = Spike added

And

$$RPD = \frac{|MSR - MSDR|}{(MSR + MSDR)/2} \; x\,100$$

where:

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR = Matrix spike duplicate recovery

Note: RPD is always expressed as a positive value.

8.5 Laboratory Control Sample Recoveries

The recoveries of each of the compounds in the LCS solution will be calculated using the following equation:

$$Laboratory\,Control\,Sample\,Recovery\ (\%R) = \left(\frac{LCSR - B}{SA}\right) x\,100$$

where:

LCSR = Concentration of target analyte in LCS
B = Concentration of target analyte in blank
SA = Concentration of spike added

9.0 QUALITY ASSURANCE/ QUALITY CONTROL

9.1 GC/MS Tuning and Performance Criteria

The GC/MS tune must be evaluated using DFTPP. The ion abundance criteria listed in Table 4, Appendix A must be met prior to any standard, blank or sample analysis. In addition, the criteria must be achieved during every 12-hour period during which standards, blanks, and samples are analyzed. The 12-hour time period for GC/MS tuning begins at the time of DFTPP injection that the laboratory submits as documentation of a compliant tune.

9.2 GC/MS Initial Calibration

All compounds including the SPCCs listed below must meet the minimum acceptable response factor (RF) of 0.05.

N-nitroso-di-n-propylamine	Hexachlorocyclopentadiene
2,4-Dinitrophenol	4-Nitrophenol

The %RSD should be less than or equal to 15% for each target analyte with the exception of the calibration check compounds (CCCs). The %RSD for the CCCs must be equal to or less than 30% for the following compounds:

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

Corrective action must be taken if any of the CCCs or SPCCs do not meet criteria. Once these criteria have been met, blanks, QC samples and environmental samples may be analyzed. Any deviations must be documented in the case narrative.

If the RSDs exceed criteria, then linearity through the origin cannot be assumed. A linear regression analysis plot not forced through "zero" may be used to calculate concentrations using area counts on the "y" axis as the dependent variable versus concentrations on the "X" axis as the independent variable. At the SERAS Laboratory, Chemstation EnviroQuant software is used . The coefficient of determination (r^2) must be greater than 0.98.

NOTE: All initial calibration standards must be analyzed prior to the analysis of any method blanks, QC samples or environmental samples.

9.3 GC/MS Continuing Calibration

After 12 hours of sample acquisition have passed, the GC/MS tune must be re-evaluated using DFTPP, and the initial calibration curve verified by analyzing a mid-level calibration standard.

1. The DFTPP must pass the criteria in Table 4, Appendix A.

2. The 50 µg/mL calibration standard must be used for the continuing calibration.

3. The %D should be less than or equal to 20% for each target analyte with the exception of the CCCs that must be equal to or less than 20% for the following compounds:

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

All compounds must meet a minimum mean RF of 0.05. For any target compounds present in the sample at a concentration greater than the RL, those analytes in the continuing calibration must meet the minimum RRF of 0.05 and the %D criteria of 20%.

The EICP area for each internal standard in the continuing calibration must be between 50% and 200% of the respective internal standard EICP area in the mid-point standard of the current initial calibration. If this criterion is not met, re-analysis is required.

4. A maximum of two continuing calibrations may be run to meet the requirements in item 3 above. A new calibration curve must be reanalyzed if both continuing calibrations are unacceptable.

   If the instrument is set up on an overnight run with two continuing calibrations back to back and the first continuing calibration passes but the second one fails, then a new initial calibration curve must be run. It is not acceptable to use the first continuing calibration if the second continuing calibration is out.

5. If any of the requirements listed in Step 3 are not met, notify the Organic Group Leader and/or Analytical Section Leader.

9.4 Internal Standard Area Evaluation

1. The amount of each internal standard in a 1 µL injection of sample extract must be 40 ng.

2. The EICP of the internal standards must be monitored and evaluated for each sample, blank, LCS, MS, and MSD.

3. If samples, blanks, LCS or MS/MSDs are analyzed immediately following an initial calibration but before another DFTPP tune and a continuing calibration, evaluation will be conducted on the basis of the internal standard areas of the 50µg/L initial calibration standard.
4. If samples, blanks, LCS or MS/MSDs are analyzed immediately following a DFTPP tune and a continuing calibration, evaluation will be conducted on the basis of the internal standard areas in the continuing calibration standard.

5. The EICP area for each internal standard in all samples, blanks, and matrix spike/matrix spike duplicates must be between 50% and 200% of the respective internal standard EICP area in the appropriate calibration standard. In addition, the retention time of each internal standard must be within 0.50 minutes (30 seconds) of its retention time in the continuing calibration standard.

6. If one or more internal standard EICP areas do not meet criteria, the GC/MS system must be inspected for malfunctions and corrections made as appropriate. When corrections are made, re-analysis of all affected samples is required.

7. If after re-analysis, the EICP areas for all internal standards meet criteria (between 50% and

200%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, only data from the analysis with EICPs within the limits are required to be submitted. If re-analysis confirms matrix effects, submit both sets of data but report the initial run.

9.5 Method Blank Analysis

A method blank is a 30 g of baked sodium sulfate (or sand) that is carried through the entire analytical procedure. The purpose of a method blank is to determine the level of contaminations associated with preparation and analysis of samples.

1. One method blank must be prepared for each batch of 20 samples.

2. A method blank should not contain more than five times the reporting limits (RL) of phthalate esters and less than the RL of the other target analytes listed in Table 1, Appendix A.

3. If a method blank exceeds the contamination limits as described above, the analytical system is considered unacceptable. The sources of contamination must be investigated so that appropriate corrective actions can be taken and documented before proceeding with any further sample analysis. All samples processed with a contaminated method blank must be re-extracted and re-analyzed. Phthalate contamination of the method blanks must be reported to the Organic Group Leader so appropriate corrective actions may be taken.

9.6 Surrogate Recoveries

The purpose of using surrogates is to evaluate the accuracy and precision associated with the preparation and analysis of samples. The recoveries of the six surrogates are calculated for all samples, blanks, and MS/MSD.

1. The surrogates are added to all samples, blanks, LCS and MS/MSD prior to extraction.

2. The surrogate recoveries are calculated using the equation in Section 8.3.

3. The client-specified surrogate recovery limits are taken from the Contract Laboratory Program (CLP) Statement of Work (revision 5/99) and are as follows:

Compound	% Recovery
Nitrobenzene-d_5	23 - 120
2-Fluorobiphenyl	30 - 115
Terphenyl-d_{14}	18 - 137
Phenol-d_5	24 - 113
2-Fluorophenol	25 - 121
2,4,6-Tribromophenol	19 - 122

4. If any two base/neutral or acid surrogates are outside QC limits or if one base/neutral or acid surrogates is below 10%, the following actions must be taken:

a. Ensure that there are no errors in calculations, surrogate solutions, and internal standards. Check that the integration of the quantitation ions of the internal standards and surrogates have been performed properly.

b. Re-analyze the sample at the discretion of the Organic Group Leader if there are no obvious errors. If a blank does not meet the specification, it may be reanalyzed alone.

c. Do not re-analyze diluted samples with a dilution ratio greater than 10.

d. If the sample associated with the MS/MSD does not meet QC limits, it should be reanalyzed only if the MS/MSD recoveries are within the limits. If the sample and the associated MS/MSD show the same pattern (i.e., outside the limits), the sample does not require re-analysis. Document in the case narrative.

NOTE: Do not re-analyze the MS/MSD, even if their surrogate recoveries fall outside the QC limits.

5. If upon re-analysis of the sample, the surrogate recoveries fall within the QC limits, then the problem was within the laboratory's control. Submit only the data from the analysis with the surrogate recoveries within the QC limits. This shall be considered the initial analysis and reported in the data package. If the re-analysis is outside the analysis holding time, both sets of data will be submitted in the data package.

6. If upon re-analysis of the sample, the surrogate recoveries still fall outside the QC limits; the sample must be re-extracted and reanalyzed as instructed by the Organic Group Leader. If the re-extraction and re-analysis of the sample solves the problem, submit only the data from the analysis with surrogate recoveries within the QC limits. This shall be considered the initial analysis and shall be reported in the data package. If the re-extraction is outside the holding time, provide the data from both analyses.

   1. If surrogate recoveries in a method blank do not meet QC limits after re-analysis, all samples associated with that blank must be re-extracted with the blank. The blank is intended to detect contamination in samples processed at the same time.

   2. If upon re-analysis of the sample associated with MS/MSD, the surrogate recoveries still fall outside the QC limits, the sample must be re-extracted.

7. If upon re-extraction and re-analysis of the sample, the surrogate recoveries fall within the QC limits, submit data only from this analysis if the holding time criteria has been met. This shall be considered the initial analysis and will be reported in the data package. If the re-extraction is outside the holding time, submit data from both analyses.

8. If upon re-extraction and re-analysis of the sample, the surrogate recoveries fall outside the QC limits, submit both sets of data. Distinguish between the initial analysis and the reanalysis in the data package.

9. Consult the Organic Group Leader before re-extracting and re-analyzing the sample.

9.7 MS/MSD Analysis

The purpose of the MS/MSD is to evaluate the accuracy and precision of the extraction and analysis, including possible sample matrix effects.

1. A MS/MSD must be analyzed every 10 samples or per project. The MS/MSD must be associated with a method blank that meets the criteria in section 9.4, a calibration in sections 9.2 and 9.3 and a tune in section 9.1. The MS/MSDs should be run on the same 12-hour shift as the sample.

2. The client-specified MS recovery limits are taken from the CLP Statement of Work (revision 5/99) and are as follows:

   Note: If the laboratory fails to meet the recovery QC limits and the RPD limits on a routine basis, the Organic Group Leader must investigate the cause and take corrective action.

Compound	% Recovery	RPD
Phenol	26 - 90	35
2-Chlorophenol	25 - 102	50
1,4-Dichlorobenzene	28 - 104	27
N-Nitroso-di-n-propylamine	41 - 126	38
1,2,4-Trichlorobenzene	38 - 107	23
4-Chloro-3-methylphenol	26 - 103	33
Acenaphthene	31 - 137	19
4-Nitrophenol	11 - 114	50
2,4-Dinitrotoluene	28 - 89	47
Pentachlorophenol	17 - 109	47
Pyrene	35 - 142	36

State in case narrative if recoveries are outside the criteria. If more than half of the spiked compounds are out, the MS/MSD should be reanalyzed. A matrix effect is indicated if the LCS data are within limits but the MS/MSD are not. A similar pattern must be observed for both the MS and MSD.

If the lab fails to meet the QC recovery limits and/or the RPD on a routine basis, the Organics Group Leader must investigate the cause and take corrective action. The MS/MSD must be prepared at the same dilution as the least diluted analysis from which sample results will be reported.

9.8 Dilution Analysis

If the concentration of any target analyte in a sample extract exceeds the initial calibration range, the sample extract must be diluted and reanalyzed as described in Section 7.6.

1. Use the results from the initial analysis to estimate the approximate dilution factor needed to bring the

highest concentration within the linear calibration range.

2. The dilution factor chosen should bring the highest target analyte within the upper half of the calibration range.

3. Submit the data from the original sample and the dilution in which analytes fall within the calibration range. If the screening procedure determines that the extract cannot be analyzed undiluted, submit the data from the first dilution and a subsequent dilution in which analytes fall within the calibration range. NOTE: Except in extreme cases, all extracts should be run undiluted to achieve the lowest detection limit.

9.9 Manual Integrations

Manual integration of all target analytes, surrogates, and internal standards will be submitted for review. The manual integration results will be flagged with a "M" and will be initialed and dated by the analyst and group leader indicating that the integration was performed properly. Documentation of the manual integration of quantitation ion peaks must be included in the data package. Refer to SERAS SOP #1001, *Chromatographic Peak Integration Procedures.*

9.10 Laboratory Control Sample

1. A LCS must be analyzed every 20 samples or per batch. The LCS must be prepared at 50 µg/L from the second source. The LCS must be associated with a method blank that meets the criteria in section 9.4, a calibration in sections 9.2 and 9.3 and a tune in section 9.1.

2. The QC limits for the LCS recoveries are listed below.

Compound	% Recovery
Phenol	70 - 130
2-Chlorophenol	70 - 130
1,4-Dichlorobenzene	70 - 130
N-Nitroso-di-n-propylamine	70 - 130
1,2,4-Trichlorobenzene	70 - 130
4-Chloro-3-methylphenol	70 - 130
Acenaphthene	70 - 130
4-Nitrophenol	70 - 130
2,4-Dinitrotoluene	70 - 130
Pentachlorophenol	70 - 130
Pyrene	70 - 130

State in case narrative if recoveries are outside criteria. On a quarterly basis, a LCS will be prepared and run that contains all of the target analytes. The above limits will be used until the first 20 points are available to prepare a control chart. At that point, control and warning limits will be calculated every 10 to 20 points and updated at least quarterly.

If the lab fails to meet the QC recovery limits on a routine basis, the Organics Group Leader and/or Analytical Section Leader must investigate the cause and take corrective action.

9.11 Initial Demonstration of Capability

Initial proficiency in SVOC analysis must be demonstrated by each analyst initially and each time significant changes are made in the procedure or for instrumentation. Each analyst will generate precision and accuracy data using a reference standard other than the source used for calibration. Four replicates of a well-mixed reference standard is analyzed using the procedures outlined in this SOP. Calculate the average mean in µg/kg and the standard deviation (s) in µg/kg. The QAO will tabulate the results from all of the analysts per matrix per parameter, and calculate control limits.

9.12 Method Detection Limit Studies

Method detection limit (MDL) studies will be run on an annual basis for the soil matrix to verify the minimum concentration that can be measured and reported with 99% confidence. A minimum of seven replicates will be used for the study (EPA 1984).

9.13 Nonconformance Memo

A nonconformance memo will be generated any time an employee notices a deficiency suspected of being a nonconformance. This nonconformance memo will be forwarded to the Quality Assurance Officer for verification of corrective action.

10.0 DATA VALIDATION

Data will be assessed in accordance with the guidelines set forth in the most current version of draft SERAS SOP #1016, *Data Validation Procedures for Routine Semi-volatile Organic Analysis*. However, data is considered satisfactory for submission when all the following requirements are met.

1. All samples must be analyzed under an acceptable tune, initial calibration, and continuing calibration check at the required frequency.

2. The QC requirements described in Section 9.0 should be met at all times. Any deviation or anomalous conditions should be discussed with the Organic Group Leader and documented on a nonconformance memo.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to EPA, Occupational Safety and Health Administration (OSHA) and corporate health and safety practices. More specifically, refer to SERAS SOP #3013, *SERAS Laboratory Safety Program* and SERAS SOP #1501, *Hazardous Waste Management*.

12.0 REFERENCES

National Environmental Laboratory Accreditation Committee (NELAC), *Quality Systems*, current approved version

U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. 1996. *Test Methods for Evaluating Solid Waste*. SW-846. 3rd ed. Method 3500B.

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U.S. Environmental Protection Agency, Contract Laboratory Program (CLP). 1999. *Statement of Work for Organic Analysis*, OLM04.2.

U.S. Environmental Protection Agency. 1984. Federal Register, 40 Code of Federal Regulations (CFR) Part 136, Appendix B, *Definition and Procedure of the Determination of the Method Detection Limit - Revision 1.11*, October 26, 1984.

13.0 APPENDICES

A - Tables
B - GPC Cleanup Procedure

APPENDIX A
Tables
SOP #1805
January 2006

Table 1. Target Compound List and Reporting Limits[1]

COMPOUND	RL[2] ,µg/kg
Phenol	333
bis(2-Chloroethyl)ether	333
2-Chlorophenol	333
1,3-Dichlorobenzene	333
1,4-Dichlorobenzene	333
Benzyl alcohol	333
1,2-Dichlorobenzene	333
2-Methylphenol	333
bis(2-Chloroisopropyl)ether	333
4-Methylphenol	333
N-Nitroso-Di-n-propylamine	333
Hexachloroethane	333
Nitrobenzene	333
Isophorone	333
2-Nitrophenol	333
2,4-Dimethylphenol	333
bis(2-Chloroethoxy)methane	333
2,4-Dichlorophenol	333
1,2,4-Trichlorobenzene	333
Naphthalene	333
4-Chloroaniline	333
Hexachlorobutadiene	333
4-Chloro-3-methylphenol	333
2-Methylnaphthalene	333
Hexachlorocyclopentadiene	333
2,4,6-Trichlorophenol	333
2,4,5-Trichlorophenol	333
2-Chloronaphthalene	333
2-Nitroaniline	333
Dimethylphthalate	333
Acenaphthylene	333
3-Nitroaniline	333
Acenaphthene	333

[1] On a wet-weight basis
[2] RL denotes Reporting Limits

Table 1. (cont'd.) Target Compound List and Reporting Limits[1]

COMPOUND	RL[2], µg/kg
2,4-Dinitrophenol	333
4-Nitrophenol	333
Dibenzofuran	333
2,6-Dinitrotoluene	333
2,4-Dinitrotoluene	333
Diethylphthalate	333
4-Chlorophenyl-phenylether	333
Fluorene	333
4-Nitroaniline	333
4,6-Dinitro-2-methylphenol	333
N-Nitrosodiphenylamine	333
4-Bromophenyl-phenylether	333
Hexachlorobenzene	333
Pentachlorophenol	333
Phenanthrene	333
Anthracene	333
Carbazole	333
Di-n-butylphthalate	333
Fluoranthene	333
Pyrene	333
Butylbenzylphthalate	333
3,3'-Dichlorobenzidine	333
Benzo(a)anthracene	333
Bis(2-Ethylhexyl)phthalate	333
Chrysene	333
Di-n-Octylphthalate	333
Benzo(b)fluoranthene	333
Benzo(k)fluoranthene	333
Benzo(a)pyrene	333
Indeno(1,2,3-cd)pyrene	333
Dibenzo(a,h)anthracene	333
Benzo(g,h,i)perylene	333

[1] On a wet-weight basis
[2] RL denotes Reporting Limits

Table 2. Characteristic Ions for Target Compounds and Surrogates

Parameter	Primary Ion	Secondary Ion(s)
1,4-Dichlorobenzene-d_4 (ISTD)[1]	152	150, 115
Phenol	94	66, 65
bis(2-Chloroethyl)ether	63	95
2-Chlorophenol	128	130, 64
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	79	77, 108
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	108	107, 90
bis(2-Chloroisopropyl)ether	45	121, 77
N-Nitroso-di-n-propylamine	70	130, 58
4-Methylphenol	107	108, 77
Hexachloroethane	117	119, 166
Naphthalene-d_8 (ISTD)[1]	136	108
Nitrobenzene	77	123, 51
Isophorone	82	138, 54
2-Nitrophenol	139	81, 109
2,4-Dimethylphenol	107	122, 77
bis(2-Chloroethoxy)methane	93	63, 123
2,4-Dichlorophenol	162	98, 164
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	102, 129
4-Chloroaniline	127	65, 129
Hexachlorobutadiene	225	190, 260
4-Chloro-3-methylphenol	107	142, 77
2-Methylnaphthalene	142	141, 115
Acenaphthene-d_{10} (ISTD)[1]	164	162, 160
Hexachlorocyclopentadiene	237	239, 95
2,4,6-Trichlorophenol	196	198, 97
2,4,5-Trichlorophenol	196	198, 97
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	77
Acenaphthylene	152	151, 76
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	65	92, 138

[1] ISTD denotes Internal Standard

Table 2. (cont'd) Characteristic Ions for Target Compounds and Surrogates

Parameter	Primary Ion	Secondary Ion(s)
Acenaphthene	153	154, 76
2,4-Dinitrophenol	184	107, 79
4-Nitrophenol	139	65, 109
Dibenzofuran	168	139
2,4-Dinitrotoluene	165	89, 63
Diethylphthalate	149	177, 105
4-Chlorophenyl-phenylether	204	141, 77
Fluorene	166	165, 163
4-Nitroaniline	65	138, 108
Phenanthrene-d_{10} (ISTD)(1)	188	187, 189
4,6-Dinitro-2-methylphenol	198	105, 121
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
Hexachlorobenzene	284	286, 142
Pentachlorophenol	266	264, 268
Phenanthrene	178	176, 76
Anthracene	178	176, 76
Carbazole	167	166, 139
Di-n-butylphthalate	149	104
Fluoranthene	202	101, 200
Pyrene	202	200, 101
Chrysene-d_{12} (ISTD)(1)	240	236, 120
Butylbenzylphthalate	149	91, 206
Benzo(a)anthracene	228	226, 229
3,3'-Dichlorobenzidine	252	254
Bis(2-Ethylhexyl)phthalate	149	167
Chrysene	228	226, 229
Perylene-d_{12} (ISTD)(1)	264	132
Di-n-Octylphthalate	149	279
Benzo(b)fluoranthene	252	126, 250
Benzo(k)fluoranthene	252	126, 250
Benzo(a)pyrene	252	126, 250
Indeno(1,2,3-cd)pyrene	276	138, 277
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	277, 138

(1) ISTD denotes Internal Standard

Table 2 (cont'd) Characteristic Ions for Target Compounds and Surrogates

Parameter	Primary Ion	Secondary Ion(s)
SURROGATES		
Phenol-d_5	99	71
2-Fluorophenol	112	64, 92
2,4,6-Tribromophenol	62	141, 143
Nitrobenzene-d_5	82	54, 128
2-Fluorobiphenyl	172	171
Terphenyl-d_{14}	244	122

Table 3. Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-Dichlorobenzene-d_4	**Naphthalene-d_8**	**Acenapthene-d_{10}**
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
bis(2-Chloroethyl) ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	bis(2-Chloroethoxy) methane	2-Nitroaniline
1,2-Dichlorobenzene	2,4-Dichlorophenol	Dimethyl Phthalate
2-Methylphenol	1,2,4-Trichlorobenzene	Acenaphthylene
Benzyl alcohol	Naphthalene	3-Nitroaniline
bis(2-Chloro-isopropyl)ether	4-Chloroaniline	Acenaphthene
4-Methylphenol	Hexachlorobutadiene	2,4-Dinitrophenol
N-Nitroso-Di-n-propylamine	4-Chloro-3-methylphenol	4-Nitrophenol
Hexachloroethane	2-Methylnaphthalene	Dibenzofuran
2-Fluorophenol (surr)	Nitrobenzene-d5 (surr)	2,4-Dinitrotoluene
Phenol-d_5 (surr)		2,6-Dinitrotoluene
		Diethyl phthalate
		4-Chlorophenyl phenyl ether
		Fluorene
		4-Nitroaniline
		2-Fluorobiphenyl (surr)

Phenanthrene-d_{10}	**Chrysene-d_{12}**	**Perylene-d_{12}**
2,4,6-Tribromophenol (surr)	Butylbenzylphthalate	di-n-Octylphthalate
4,6-Dinitro-2-methylphenol	3,3'-Dichlorobenzidine	Benzo(b)fluoranthene
N-Nitrosodiphenylamine	Benzo(a)anthracene	Benzo(k)fluoranthene
4-Bromophenyl phenyl ether	bis(2-Ethylhexyl) phthalate	Benzo(a)pyrene
Hexachlorobenzene	Chrysene	Indeno(1,2,3-cd) pyrene
Pentachlorophenol	Terphenyl-d14 (surr)	Dibenzo(a,h)anthracene
Phenanthrene		Benzo(g,h,i)perylene
Carbazole		
Anthracene		
di-n-butylphthalate		
Fluoranthene		
Pyrene		

Table 4. Ion Abundance Criteria for Tune (DFTPP) [1]

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 abundances of m/z 442 may be up to 110 percent that of m/z 198.

[1] Criteria taken from U.S. EPA CLP, 1999.

APPENDIX B
GPC Cleanup Procedure
SOP #1805
January 2006

GPC CLEANUP PROCEDURE

1. Calibrate the GPC instrument by injecting 10 µL of working GPC standard. The pump flow rate should be set at 5 mL/minute. Elute with methylene chloride and collect the fraction between bis(2-ethylhexyl)phthalate and perylene to establish the collection time window.

2. Once the collection time window is established, inject a methylene chloride blank to make sure that all calibration components have eluted from the column.

3. Dilute the final BNA extracts to 4 mL with methylene chloride for samples, the method blank, the LCS and the MS/MSD.

4. Before injecting the samples, pass each extract (4-mL volume) through a Gelman, 0.45µm Acrodisc CR PTFE filter into separate clean 4-mL vials.

5. Load all these vials containing the filtered extracts on the autosampler and start the sequence.

6. Collect the GPC-cleaned extracts from the fraction collector, transfer to concentrator tubes and concentrate the extracts to a final volume of 0.5 mL using the TurboVap.

7. Proceed with GC/MS analysis by adding appropriate amount of internal standards to the final 0.5-mL extracts.