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MOBILE, HIGH HAZARD, GC/MS LABORATORY OPERATION – ANALYSIS FOR CREOSOTE/PCP IN SOIL

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to describe the operation of the mobile high hazard gas chromatography/mass spectroscopy (GC/MS) laboratory for the analysis of non-aqueous samples. To SERASh the sensitivity and detection limits of a flame ionization detector (FID), the mass spectrometer is run in the Selective Ion Monitoring (SIM) mode. The target compounds are creosote compounds and pentachlorophenol (PCP), listed by the SIM group in Appendix A. The quantitation limit of each analyte is listed in Appendix B.

2.0 METHOD SUMMARY

Ten grams (g) of sample are spiked with the surrogate compounds 2-fluorobiphenyl and 2,4,6-tribromophenol mixed with approximately 10 g anhydrous sodium sulfate, and extracted three times with 20:80 acetone: methylene chloride solution. The three extracts are combined, and 1.0 mL is aliquoted for analysis. Before injection on the mass spectrometer, internal standard mix is spiked into the 1.0 mL extract.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Storage

Sample extracts must always be protected from light and kept in the refrigerator at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

All unused portions of samples will be maintained in the shipping coolers until disposal or return to the site.

3.2 Holding Times

Extraction of soil/sediment samples should be completed within seven days of sampling. Samples which have undergone treatment should be extracted with seven days of sample receipt.

Soil/sediment sample extracts must be analyzed within 40 days of sample extraction.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

A disadvantage of using the GC/MS in the SIM mode is that a complete characterization of the sample extract normally performed using linear scan and library search capabilities is not available here. In the process of dwelling on only the specific ions required to identify and quantify the compounds of interest (used to enhance the method detection limit), this library search function is sacrificed.

There may be down-time associated with electrical spikes/power outages due to thunderstorms in the site area. This may result in the loss of a day of work. The appropriate people (i.e., SERAS management, the Work Assignment Manager, etc.) will be notified by the field analyst if this occurs.

The lab is not equipped with the facilities to extract water, drum liquid, or any aqueous samples, nor can any samples be concentrated.



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5.0 EQUIPMENT/APPARATUS

The following equipment/apparatus are required:

- Spatula, stainless steel or teflon
- Vials and caps, 2-mL for GC autosampler
- Balance - analytical capable, of accurately weighing ± 0.001 g
- Screw top jar, 4-oz.
- Shaker table - orbital, capable of 250 revolutions per minute (rpm)
- Serum vials, 100-mL, with crimp-top seals
- Hewlett-Packard 5971A GC/MS - An analytical system complete with gas chromatograph and all required accessories including syringes, analytical columns, gases, a mass selective detector, an autosampler, and controlled by Chem Station software. Analytical column is:

Rtx - 5 - SE-54 equivalent - 30 m x 0.25 mm ID, 0.5 μ m film thickness

Note: With this system, the temperatures in the source, analyzer and mass spectrometer are controlled by the transfer line interface. The temperature of the transfer line is set, and the resulting temperature of the mass spectrometer's parts are approximated. For a detailed explanation of this, and or the actual temperatures of the mass spectrometer parts, consult the instrument hardware manual.

6.0 REAGENTS

1. Solvent is a mixture of 20:80 acetone/methylene chloride of pesticide residue analysis grade or equivalent.
2. Sodium sulfate anhydrous powder. Heated at 400°C for four (4) hours, cooled in a dessicator and stored in a tight glass jar.
3. A 50 μ g/mL of Decafluorotriphenylphosphine (DFTPP) is prepared in methylene chloride.
4. Surrogate Spike Mix:

Surrogate standards are added to all samples, blanks, and matrix spikes prior to extraction. A 1000 μ g/mL solution of 2-fluorobiphenyl and 2,4,6-tribromophenol in methylene chloride is prepared from neat compounds. Store the spiking solution at 4°C ($\pm 2^\circ$ C) in Teflon-sealed containers and protect from light. The solutions should be checked frequently for signs of degradation or evaporation. These solutions must be replaced after 12 months, or sooner if comparison with quality control check samples indicates a problem.

5. Internal Standard Mix:

Prepare the internal standard mix by diluting a 2000 μ g/mL solution (Supelco Catalog Number 4-8902 or equivalent) of the following compounds to 200 μ g/mL in methylene chloride:

Acenaphthene - d₁₀



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

6. Matrix Spike/ Matrix Spike Duplicate Spiking Mixture:

Prepare a spiking solution in methylene chloride from neat compounds that contains the following compounds at the listed concentrations. Store the spiking solution at 4°C (±2°C) in Teflon-sealed containers and protect from light. The solution should be checked frequently for signs of degradation or evaporation. These solutions must be replaced after 12 months, or sooner if comparison with quality control check samples indicates a problem.

naphthalene	1000 µg/mL
pentachlorophenol	2000 µg/mL
benzo(a)pyrene	1000 µg/mL

7. Calibration Standards:

Prepare calibration standards at five concentration levels (0.5, 1.0, 5.0, 10.0 and 25.0 µg/mL). Each calibration standard should contain each compound of interest and each surrogate. Great care must be taken to maintain the integrity of all standard solutions. Store all standard solutions at 4°C (±2°C) and protect from light. Fresh standards should be prepared every six months at a minimum. Prepare the stock solution by adding a solution containing 2000 µg/mL each of 2-fluorobiphenyl, 2,4,6-tribromophenol, carbazole, and pentachlorophenol to a commercially available mixture containing 2000 µg/mL (Supelco Catalog #4-8905 or equivalent) of each of the following compounds:

1. Naphthalene
2. Acenaphthylene
3. Acenaphthene
4. Fluorene
5. Phenanthrene
6. Anthracene
7. Fluoranthene
8. Pyrene
9. Benzo(a)anthracene
10. Chrysene
11. Benzo(b)fluoranthene
12. Benzo(k)fluoranthene
13. Benzo(a)pyrene
14. Dibenzo(a,h)anthracene
15. Benzo(g,h,i)perylene
16. Indeno(1,2,3,c,d)pyrene



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Then the mixture of all 20 compounds will be diluted to the needed concentrations.

7.0 PROCEDURE

7.1 Sample Preparation and Extraction

1. Transfer the sample container into the glove box. Open the sample bottle and discard any foreign objects such as sticks, leaves, and rocks. Mix the sample thoroughly.
2. Weigh approximately 10 g of sample to the nearest 0.1 g into a tared 100-mL serum bottle and add 10-20 g of anhydrous sodium sulfate. Mix well. The sample should have a sandy texture at this point. A method blank must be prepared by using 10 g of sand (or baked sodium sulfate) according to the same procedure at the frequency of one per 20 samples.
3. The sample for total solids is weighed in conjunction with the sample for the extraction. The total solid for the MS/MSD is based on the corresponding sample. The blank is expected to be 100% solid.

Weigh and record the aluminum sample dish to the nearest .01-gm. Weigh at least 10 g of the soil/sediment into the aluminum dish. Determine the total percent solid by drying in an oven placed inside a fume hood overnight at 105°C. Before weighing, cool in a desiccator. Concentrations of individual analytes will be reported relative to the dry weight of the sediment. Calculate the total percent solid using the following equation:

$$\% \text{Total Solids} = \frac{\text{Weight of Dried Sample with Dish (g)} - \text{Dish Weight (g)}}{\text{Weight of Wet Sample with Dish (g)} - \text{Dish Weight (g)}} \times 100$$

4. Weigh two additional 10-g portions of samples to the nearest 0.1-g for use as matrix and matrix spike duplicates (MS/MSD) at a rate of one per ten samples or 10%.

NOTE: The sample may be specified on a Chain of Custody record for this purpose.

5. Add 500 µL of the surrogate spiking solution to the method blank(s), the MS/MSD pair(s) and all the samples. The resulting concentration is 5 ppm in the final extract analyzed by the GC/MS.
6. Add 40 mL of 80:20 methylene chloride:acetone to the sample; crimp and secure on the shaker table. Shake the sample for 30 minutes at approximately 250 revolutions per minute (rpm).
7. Decant the solvent into a 4 oz. screw top jar.
8. Repeat steps 6 and 7 using 30 mL methylene chloride:acetone mixture. Combine all 100 mL of the three solvent mixtures from each of the extractions into the 4 oz. jar.

If the samples contain an unusual amount of silt, centrifuge a 10 mL portion of the extract before



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aliquoting 1 mL for analysis.

If the sample appears to be oily or emits a strong odor detectable when employing a hood, high concentrations of creosote are likely to be present. Advise the analyst for determination of dilution of the sample prior to aliquoting.

9. Aliquot 1.0 mL of the 100 mL combined extract into an autosampler vial. Spike with 20 μ L of 200 ppm internal standard mixture. The concentration of each internal standard is 4.0 ppm in the 1.0 mL extract.
10. Only 10 mL of the extract is saved for future disposal on the site of the sampling, and discard the remaining 90 mL in appropriate solvent pail.
11. Dispose of the extracted soil/sediment that is in the 100 mL serum vial into the disposal drums labeled "Lab Scraps" at the site.

7.2 Instrument Conditions

The conditions listed below are used for standards and sample analysis.

Column	Restek Rtx-5(crossbonded SE-54) 30 meter x 0.25 mm ID, 0.50 μ m film thickness
Injector Temperature	290°C
Transfer Line Temperature	315°C
Temperature Program	55°C for 2 min 10°C/min to 295°C hold for 5 min 25°C/min to 305°C hold for 8 min
Splitless Injection	Split time = .88 min
Injection Volume	2 μ L
Timed EMV Program	Delta EMV Time 506 15:00 400 21:50
Column Flow	0.95 mL/min, EPC enabled

7.3 Tune (DFTPP)

The instrument must be turned to meet the ion abundance criteria listed in Appendix C for a 50-ng (1 μ L) injection of DFTPP. This criteria must be demonstrated every 24 hours during analysis.

7.4 Initial Calibration

1. Add 20 μ L of internal standard solution to each 1-mL aliquot of calibration standards.
2. Inject 2 μ L each of the calibration standards after a successful DFTPP injection.



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3. Calculate and tabulate the relative response factor (RRF) against the concentration for each compound, including the surrogates, by using the equation listed 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 for the compound to be measured
- C_{IS} = Concentration of the internal standard (ng/ μ L)
- A_{IS} = Area of the characteristic ion for the internal standard.
- C_x = Concentration of the compound to be measured (ng/ μ L)

The % RSD of the RRF for each analyte must be less than or equal to 30%. The average RRF of each compound must not be less than 0.05.

7.5 Continuing Calibration

A check of the initial calibration curve must be performed every 24 hours during analysis.

1. Inject 2 μ L of a 5.0 μ g/mL standard containing internal standards.
2. Calculate and tabulate the daily RRF for each compound. All daily RRF must be equal to or greater than 0.05.
3. Calculate the percent difference (% D) of each daily RRF compared to the average RRF from the initial calibration curve. The % D for all compounds can be calculated using the equation listed below and must be less than or equal to 25%.

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

4. All sample and standards are quantitated using the average response factors from the calibration range, NOT the daily calibration check.

7.6 Sample Analysis

Sample extracts may be analyzed only after the GC/MS system has met the DFTPP, initial calibration, and continuing calibration requirements mentioned above. The same instrument conditions must be employed for the analysis of samples as were used for calibration.

1. Add 20 μ L of the internal standard solution into the method blank, the MS/MSD, and all the



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sample extracts.

2. Inject 2 μL each of the method blank, the MS/MSD, and all the sample extracts.
3. If the analyst has reason to believe that diluting the final extracts will be necessary, an undiluted run may not be required.
4. If analytes are detected at a level greater than the highest calibration standard, sample extracts must be diluted so that the analyte response is within the linear range established during calibration.
5. If dilutions of sample extracts are made, additional internal standards must be added to maintain the required concentration (4.0 ng/ μL) of each internal standard in the extract.

7.7 Identification of Target Compounds

Target compound identification will be conducted by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound. Two criteria must be satisfied to verify the identifications:

- Elution of the sample component at the GC relative retention time as the standard component
 - Correspondence of the sample component and standard component mass spectra
1. For establishing correspondence of the GC relative retention time (RRT), the sample component RRT must compare within ± 0.06 RRT units of the RRT of the standard component. If coelution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the component of interest.
 2. For comparison of standard and sample component mass spectra, reference mass spectra must be obtained from the 5.0 $\mu\text{g}/\text{mL}$ standard. These standard spectra may be obtained from the run used to obtain reference RRTs.
 3. The requirements for qualitative verification by comparison 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 in the spectrum equals 100%) must be present in the sample spectrum.
 - b. The relative intensities of ions specified in (a) must agree within $\pm 20\%$ between the standard and sample spectra. (For example: for an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 30-70%.)



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- c. Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst making the comparison. All compounds meeting the identification criteria must be reported with their spectra. For all compounds below the quantitation limit, report the actual value followed by "J", e.g., "3J".
4. If a compound cannot be verified by all of the criteria in step 3, but in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the analyst shall report that identification and proceed with the calculation in Section 8.0. The analyst should note in the case narrative that technical judgment was utilized.

8.0 CALCULATIONS

8.1 Target Compounds

Identified target compounds must be quantitated by the internal standard method.

Calculate the concentration in the sample using the relative response factor (RRF) obtained from the initial calibration standard as determined in 7.4 and the equation listed below.

$$\text{Concentration } (\mu\text{ /kg}) = \frac{A_X \cdot C_S \cdot V_T \cdot DF}{A_{IS} \cdot RRF_{avg} \cdot W \cdot V_I}$$

where:

- A_X = Area of the characteristic ion for the compound to be measured
- I_S = Amount of internal standard injected (ng)
- V_T = Volume of the concentrated extract (μL)
- DF = Dilution factor
- A_{IS} = Area of the characteristic ion for the internal standard
- RRF = Relative response factor
- W = Weight sample (g)
- V_I = Volume of extract injected (μL)

When the target compound concentrations are below the quantitation limits but the spectrum meets the identification criteria, report the concentration as estimated by flagging the results with a "J".

8.2 Percent Solids

Percent solids are calculated using the following equation:

$$\text{PercentSolids} = \frac{\text{TWD} - \text{DW}}{\text{TW} - \text{DW}} \times 100$$

where:



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TWD = total weight of dried sample and dish
DW = dish weight
TW = total weight of wet sample and dish

8.3 Surrogate Spike Recoveries

Calculate surrogate standard recovery on all samples, blanks, and spikes by using the equation listed below.

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

where:

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

8.4 Matrix Spike Recoveries

The percent recoveries and the relative percent difference (RPD) between the recoveries of each of the 11 compounds in the matrix spike samples will be calculated and reported by using the following equations:

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

where:

SSR = Spike sample result
SR = Sample result
SA = Spike added

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

where:

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

The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

8.5 Relative Response Factor

The relative response factor (RRF) for each specific analyte is quantitated based on the following equation :



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

where:

- A_x = Area of the characteristic ion for the compound to be measured
- C_{is} = Concentration of the internal standard (ng/ μ L)
- A_{is} = Area of the characteristic ion for the internal standard
- C_x = Concentration of the compound to be measured (ng/ μ L)

The average response factor (ARF) is calculated from the average of the five points on the calibration range.

The sample concentration will be corrected for % solids.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

9.1 Tune (DFTPP)

Prior to initiating any data collection activities involving samples, blanks, or standards, it is necessary to establish that a given GC/MS system meets the instrument tune criteria specified in Appendix C. The purpose of this instrument check is to assure correct mass calibration, mass resolution, and mass transmission. This is accomplished through the analysis of DFTPP.

1. The analysis of DFTPP must be performed every 24 hours during the analysis.
2. The key ions produced during the analysis of DFTPP and their respective ion abundance criteria are given in Appendix C.

9.2 Initial Calibration for Target Compounds and Surrogates

Prior to the analysis of samples and required blanks, and after instrument performance criteria have been met, the GC/MS system must be initially calibrated at a minimum of five concentrations to determine the linearity of response utilizing target compound and surrogate standards.

1. The levels of the initial calibration standards for creosote/PCP target compounds and surrogates are 0.5, 1.0, 5.0, 10.0 and 25.0 μ g/mL.
2. The calibration of the GC/MS is evaluated on the basis of the magnitude and stability of the relative response factors of each target compound and surrogate. The minimum RRF of each compound at each concentration level in the initial calibration across all five points must be equal to or greater than 0.05; the %RSD must not exceed 30%.

9.3 Continuing Calibration for Target Compounds and Surrogates



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Once the GC/MS system has been calibrated, the calibration must be verified each 24-hour time period for each GC/MS system during the analysis.

1. The level of the continuing calibration standard for target compounds and surrogates is 5.0 µg/mL.
2. The standard is to be analyzed every 24 hours after an acceptable DFTPP analysis.
3. The continuing calibration of the GC/MS system is evaluated on the basis of the magnitude of the relative response factors and the percent difference between the average RRF of each compound from the initial calibration and the RRF of that compound in the continuing calibration standard. The minimum RRF of each compound in the continuing calibration must be greater than or equal to 0.05. The %D must not exceed 25%.
4. If any of the requirements listed in Item 3 are not met, another initial calibration must be analyzed.

9.4 Method Blank Analysis

A method blank is a volume of a clean reference matrix (10 g clean sand or baked sodium sulfate) that is carried through the entire analytical procedure. The volume of the reference matrix must be approximately equal to the volume of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

1. The method blank must be prepared for each batch not exceeding 20 samples.
2. The method blank must contain less than or equal to the quantitation limit (QL) of any single target compound.
3. If a method blank exceeds the limits for contamination above, the analyst must consider the analytical system out of control. The source of the contamination must be investigated and appropriate corrective actions taken and documented before further sample analysis proceeds. All samples processed with a contaminated method blank must be re-extracted and reanalyzed.

9.5 Surrogate Recoveries

The recoveries of the two surrogates are calculated from the analysis of each sample, blank, matrix spike and matrix spike duplicate. The purpose of the surrogates is to evaluate the preparation and analysis of samples.

1. The surrogates are added to each sample, blank, matrix spike, and matrix spike duplicate prior to extraction, at the concentrations described in Sections 6.0 and 7.1.
2. The recoveries of the surrogates are calculated according to the equation in Section 8.3.



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- The advisory quality control limits are given below. No action is required at this time if the surrogates do not meet the QC advisory limits. However, frequent failures to meet the limits for surrogate recovery warrant investigation by the laboratory.

<u>Compound</u>	<u>% Recovery</u>
2-Fluorobiphenyl	30 - 115
2,4,6-Tribromophenol	19 - 122

9.6 Matrix Spike and Matrix Spike Duplicate Analysis

The purpose of spiking target compounds into two aliquots of a sample is to evaluate the effects of the sample matrix on the methods used in this SOP.

- The MS/MSD must be prepared every 20 samples per matrix within each project.
- The mixture of the spike solution specified in Section 6.0 must be used to result in the concentration specified in Section 7.1.
- The recoveries of the matrix spike compounds are calculated according to the equation in Section 8.4. The relative percent difference between the results for each spiked analyte of the matrix spike and the matrix spike duplicate is calculated according to the equation in Section 8.4.
- The advisory quality control limits for recovery and relative percent difference are given below. These limits are only advisory at this time, and no further action is required when the limits are exceeded.

<u>Compound</u>	<u>% Recovery</u>	<u>RPD</u>
Naphthalene	46 - 118	31
Pentachlorophenol	9 - 103	50
Benzo(a)pyrene	26 - 127	31

- Analysts may substitute and analyze a blank MS/MSD if the soil samples appear to be highly concentrated or if there are no suitable soil samples for MS/MSD extraction.

9.7 Dilution Analysis

If the concentration of any sample extract exceeds the initial calibration range, that sample extract must be diluted and reanalyzed as described in Section 7.6, steps 4 and 5.

- Use the results of the original analysis to determine the approximate dilution factor required to get the largest analyte peak within the initial calibration range.
- The dilution factor chosen should keep the response of the largest analyte peak for a target



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compound in the upper half of the initial calibration range of the instrument.

3. Do not submit data for more than two analyses, i.e., the original sample and one dilution, or, from the most concentrated dilution analyzed and one further dilution.

10.0 DATA VALIDATION

Data validation will be performed by the Data Validation and Report Writing Group and therefore it is not applicable to this SOP. However, data is considered satisfactory for submission purposes when ALL the requirements mentioned below are met.

1. All samples must be analyzed under an acceptable tune, initial calibration, and continuing calibration check at the required frequency.
2. All the QC requirements described in Section 9.0 must be met all the time except advisory limits for surrogates and MS/MSD recoveries.

11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, refer to U.S. EPA, OSHA and corporate health and safety practices. More specifically, refer to ERT/SERAS SOP #3013, SERAS Laboratory Safety Program.

12.0 REFERENCES

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Selective Ion Monitoring (SIM) Groups

<u>Compound Name</u>	<u>Quant Ion</u>	<u>Ions in group</u>
<u>Group 1: 10.0 to 15.0 min</u>		
d8-naphthalene [ISTD]	136	68, 108, 127, 128, 129
naphthalene	128	136, 151, 171, 172
2-fluorobihpenyl {SURR}	172	
<u>Group 2: 15.00 to 18.8 min</u>		
d10-acenaphthene [ISTD]	164	151, 152, 153, 160, 162,
acenaphthene	153	164, 165, 166, 167, 143
fluorene	166	330, 332,
2,4,6-tribromophenol {SURR}	330	
<u>Group 3: 18.8 to 21.5 min</u>		
Pentachlorophenol	266	80, 94, 166, 167,
d10-phenanthrene [ISTD]	188	168, 176, 178, 179, 188
phenanthrene	178	264, 266, 268
anthracene	178	
carbazole	167	
<u>Group 4: 21.50 to 28.0 min</u>		
fluoranthene	202	100, 101, 120, 202, 226
pyrene	202	228, 229, 236, 240
benzo(a)anthracene	228	
d12-chrysene [ISTD]	240	
chrysene	228	
<u>Group 5: 28.0 min to end of run</u>		
benzo(b)fluoranthene	252	126, 250, 252, 253, 253.20
benzo(k)fluoranthene	252	260, 264, 265
benzo(a)pyrene	252	
d12-perylene [ISTD]	264	

[ISTD] denotes internal standard for group
{SURR} denotes surrogate



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APPENDIX B
Target Compound List and Quantitation Limits
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Target Compound List and Quantitation Limits

COMPOUND	QL ⁽¹⁾ (mg/kg)
Acenaphthylene	5
Naphthalene	5
Acenaphthene	5
Fluorene	5
Pentachlorophenol	5
Phenanthrene	5
Anthracene	5
Carbazole	5
Fluoranthene	5
Pyrene	5
Benzo(a)anthracene	5
Chrysene	5
Benzo(b)fluoranthene	5
Benzo(k)fluoranthene	5
Benzo(a)pyrene	5
Indeno(1,2,3,-cd)pyrene	5
Dibenzo(a,h)anthracene	5
Benzo(g,h,i)perylene	5

(1) QL denotes Quantitation Limits



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APPENDIX C
Ion Abundance Criteria for Tune (DFTPP)
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Ion Abundance Criteria for Tune (DFTPP)

<u>Mass</u>	<u>Ion Abundance Criteria</u>
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.