# Semivolatile Organic Compounds By Gas Chromatography / Mass Spectrometry (GC/MS)

References: **EPA 8270D**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision IV, February 2007.

**EPA 8000C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision III, March 2003.

## 1. Scope and Application

**Matrices:** Solid, waste and waste water, soil, sediment, tissue and ground water.

**Definitions:** Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification of acidic, neutral and basic organic compounds soluble in methylene chloride. The extraction and cleanup methods that are typically used for this method are listed in Section 10.3.1. Please see the appropriate extraction SOP(s) for further information. The individual target compounds are found in Section 16, Table D, and include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, nitrosamines, aldehydes, ethers, anilines, pyridines, aromatic nitro compounds and phenols, including nitro phenols.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

## 2. Summary of Method

Aqueous samples are extracted with methylene chloride in a 2 Liter Separatory Funnel (Method 3510) at the required pH. Soil samples are generally extracted by Microscale Solvent Extraction (Method 3570), however other extraction methods can be applied. The extract is concentrated to its final volume in preparation for analysis, or may now undergo cleanups.

Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS). Identification of target analytes is accomplished by

comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

### 2.1 Method Modifications from Reference

None.

## 3. Reporting Limits

Analytes determined are measured in the range of 0.5 to 25ug/L for water samples, and 33.3 to 1667ug/Kg for soils, sediments, and solids. Tissue samples are measured in the range of 100 to 5000ug/Kg. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed. Analytes detected over these concentration ranges will be diluted and re-analyzed for accurate quantification.

## 4. Interferences

**4.1** Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

**4.2** The injection port of the gas chromatograph can become contaminated with high boiling compounds resulting in the loss of sensitivity of Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, Benzaldehyde, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 4-chloroaniline, benzyl alcohol, 2,4-Dinitrotoluene and 2, 3, and 4-Nitroaniline. It may be necessary to replace the injection port liner routinely to prevent this loss of sensitivity. Clipping off approximately four inches of the column at the injection end may also increase sensitivity of the phenolic compounds. Low instrument response can be detected during the daily tuning procedure by including pentachlorophenol and benzidine in the daily tuning mix.

**4.3** Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

**4.4** Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the *Reagent, Solvent and Standard Control* SOP (G-008) and *Laboratory Glassware Cleaning SOP* (G-002), for additional details.

**4.5** Many interferences can be removed by sample cleanup. The cleanup methods performed include those listed in Section 10.3.1. Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest.

**4.6** Benzidine and pyridine may be subject to oxidative losses during solvent concentration and demonstrate poor chromatographic behavior.

**4.7** Hexachlorocyclopentadiene and pyridine are subject to thermal decomposition in the injection inlet, and hexachlorocyclopentadiene is subject to chemical reaction with acetone, and photochemical decomposition.

**4.8** N-nitrosodimethylamine is difficult to separate from the solvent front under the chromatographic conditions described within.

N-nitrosodiphenylamine decomposes in the GC inlet and cannot be separated from diphenylamine.

# 5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

*Solid samples:* A minimum of 100 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

*Water samples:* A minimum of 2 liters of sample must be collected in amber glass bottles.

*Tissue samples:* A minimum of 20 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

## 6.2 Sample Preservation

*Solid samples:* The samples must be refrigerated and maintained at 4±2°C until extraction and analysis. Sediment samples can be frozen at -20±5°C until extraction to extend hold time. The extracts must be refrigerated and maintained at 4±2°C until analysis.

*Water samples:* The samples must not be preserved except by refrigeration at 4±2°C until extraction and analysis. The extracts must be refrigerated and maintained at 4±2°C until analysis.

*Tissue samples:* The samples must be stored frozen at -20±5°C until extraction. The extracts must be refrigerated and maintained at 4±2°C until analysis.

## 6.3 Sample Shipping

No special shipping requirements.

## 6.4 Sample Handling

*Solid samples:* All solid samples must be extracted within 14 days from the date of collection. Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

*Water samples:* All water samples must be extracted within 7 days from the date of collection. Sample extracts must be analyzed within 40 days from date of extraction. Sediment samples can be frozen to extend hold time.

*Tissue samples:* Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

# 7. Equipment and Supplies

**7.1 Gas chromatograph:** Programmable, heating range from 40C to 350C; split-splitless-type inlet system, (Agilent 6890); mass selective detector (Agilent 5973); automatic injector (Agilent 7683 or similar).

**7.2 Chromatography Column:** Fused silica capillary column, 0.25mm ID x 30m length, .25um film thickness (Zebron ZB-SemiVolatiles, Phenomenex Corporation, 5% Polysilarylene - 95% Polydimethylsiloxane , or equivalent).

**7.3 Data Acquisition System:** Computerized system for collecting, storing, and processing detector output (Hewlett Packard Enviroquant target software) or equivalent.

**7.4 Gases:** Ultra high purity BIP helium (99.9995%); Compressed nitrogen for N-Evap.

**7.5 Syringes:** 10uL to 1.0mL.

**7.6 Glass:** Class A volumetric glass with varying volumes

**7.7 GC Injection Port Liner**: Phenomenex Direct Connect Top Hole or Phenomenex Focus Liner

# 8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

**8.1 Methylene Chloride:** ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

**8.2 Acetone:** ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

**8.3 Hexane:** ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

**8.4 Methanol:** ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

**8.5 Analytical Standards:** Standards should be stored at –10°C or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or if breakdown is observed. Stock standards are given an 1 year expiration from the date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a 6 months expiration from the preparation date or the expiration of the primary solution whichever occurs first.

**8.5.1 Surrogates:** Base/Neutral stock surrogate (5000ug/mL) – includes Nitrobenzene-d5, 2-Fluorobiphenyl, and Terphenyl-d14. Acid stock surrogate (10000 ug/mL) - includes Phenol-d5, 2-Fluorophenol, and 2,4,6-Tribromophenol. Restek, Ultra Scientific, or equivalent.

**8.5.1.1** To prepare the surrogate spiking solution mix for 8270: Add 2mL of the base neutral mix (5000 ug/mL) and 1.0mL of the acid surrogate mix (10000 ug/mL) to a 500mL volumetric flask. Dilute to volume with Acetone to provide a 20 ug/mL surrogate solution.

**8.5.2** **Field Surrogates:** Fluoranthene-d10 (2000ug/mL), and Benzo(a)pyrene D12 (200ug/mL) is commercially obtained from Chem Service**.**
NOTE: These surrogates are not for use with "routine" 8270 analyses, but are a project specific requirement for the analysis of SVOC in a PUF matrix.

**8.5.2.1** To prepare the Field surrogate spiking solution, add 5mL of Benzo(a)pyrene-d12 (200ug/mL), and 0.5mL of Fluoranthene-d10 (2000ug/mL) to a 1mL volumetric flask. Dilute with methylene chloride to provide a 100ug/mL spiking solution.

**8.5.3** **Stock Calibration Mix Standards:** Pre-made, commercially available solutions.

- PAH Mix - ECS-A-032 (2000ug/mL)
- EPA 8270 Add-ons - ECS-A-031 (2000ug/mL)
- EPA 625 B/N Mix#1 - ECS-A-030 (2000ug/mL)
- Benzidines Mix - ECS-A-007 (2000ug/mL)
- Phenols Mix - ECS-A-006 (2000ug/mL)
- Custom Semivolatiles Mix (containing acetophenone, atrazine, benzaldehyde, biphenyl, and caprolactam) – Restek – cat # 57685 (2000ug/mL)
- 1,2,4,5-Tetrachlorobenzene – Accustandard (1000ug/mL)
- 2,3,4,6-Tetrachlorophenol – UltraScientific (2000ug/mL)
- Pentachloronitrobenzene – Ultra Scientific (1000ug/mL)
- 2-Chloroaniline – Ultra Scientific (NEAT)
  - ~20mg of NEAT standard is diluted to 10mL in DCM to create a stock solution ~2000ug/mL for use in preparing the ICAL stock
- Benzo(a)pyrene-D12 – Cambridge Isotope Laboratory
  - ~20mg of NEAT standard is diluted to 10mL of DCM to create a stock solution ~2000ug/mL for use in preparing the ICAL stock

The above surrogate stock mixes are also added to the standard mixture. Solutions equivalent to Environmental Consulting & Supplies (ECS) are acceptable. Due to the highly reactive nature of Benzidine and Benzaldehyde and their tendency to degrade when mixed with all other target analytes found in this method, two separate ICAL solutions are prepared.

**8.5.3.1** Preparation of the first stock standard (100ug/mL) to ***10mL*** stock solution in methylene chloride is as follows:

**8.5.3.1.1** EPA 8270 Add-ons (2000ug/mL): 0.5mL

**8.5.3.1.2** EPA 625 B/N Mix #1 (2000ug/mL): 0.5mL

**8.5.3.1.3** Phenols Mix (2000ug/mL): 0.5mL

**8.5.3.1.4** PAH Mix (2000ug/mL): 0.5mL

**8.5.3.1.5** Acid Surrogate (10000ug/mL): 100uL

**8.5.3.1.6** B/N Surrogate (5000ug/mL): 200uL

**8.5.3.1.7** 1,2,4,5-Tetrachlorobenzene (1000ug/mL): 1.0mL

**8.5.3.1.8** 2,3,4,6-Tetrachlorophenol (2000ug/mL): 0.5mL

**8.5.3.1.9** 2-chloroaniline Stock (~2000ug/mL): ~0.5mL

**8.5.3.1.10** Benzo(a)pyrene-d12 Stock (~2000ug/mL): ~0.5mL

**8.5.3.1.11** Fluoranthene-d10 (2000ug/mL): 0.5mL

**8.5.3.1.12** Pentachloronitrobenzene (1000ug/mL) : 1.0mL

**8.5.3.2** Preparation of the second stock standard (100ug/mL) to 10mL in methylene chloride is as follows:

**8.5.3.2.1** Benzidines Mix (2000ug/mL): 0.5mL

**8.5.3.2.2** Custom Semivolatiles Mix (2000ug/mL): 0.5mL

**8.5.3.3** Preparation of calibration levels using the above stock solutions. All eight levels are prepared using the first stock solution. Only levels 1-7 are prepared using the second stock solution.

**8.5.3.3.1** **Level 8 (50ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 1.0mL in methylene chloride.

**8.5.3.3.2** **Level 7 (25ug/mL):** 2.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride

**8.5.3.3.3** **Level 6 (15ug/mL):** 1.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

**8.5.3.3.4** **Level 5 (10ug/mL):** 1.0mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

**8.5.3.3.5** **Level 4 (5.0ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

**8.5.3.3.6** **Level 3 (2.0ug/mL):** 0.20mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

**8.5.3.3.7** **Level 2 (1.**0ug/mL): 0.1mL of Calibration Stock diluted to a final volume of 10mL in Methylene chloride.

**8.5.3.3.8** **Level 1 (0.5ug/mL):** 0.05mL of Calibration stock diluted to a final volume of 10mL in methylene chloride.

**8.5.4 Initial Calibration Verification (ICV):**

**Initial Calibration Verification primary solutions (200ug/mL):**
Custom BNA Spike – Commercially available NSI – PN Q3461.

1,2,4,5-Tetrachlorobenzene solution (2000ug/mL): Ultra Scientific
Pentachloronitrobenzene solution (5000ug/mL): Supelco
2-Chloroaniline solution (2000ug/mL): Supelco
Benzoic Acid Solution (2000ug/mL): Restek
Benzaldehyde Solution (2000ug/mL): Restek
Benzidine solution (1000ug/mL): Restek

**8.5.4.1 Initial Calibration Verification solution (10ug/mL):** 0.50mL of Custom BNA Spike diluted to a final volume of 10mL in methylene chloride.

**8.5.4.2 Initial Calibration Verification solution** for 1,2,4,5-Tetrachlorobenzene, 2-Chloroaniline, Pentachloronitrobenzene, and Benzoic Acid (10ug/mL): 0.5mL of 8270 LCS2 diluted to a final volume of 10mL in Methylene chloride.

**8.5.4.3 Initial Calibration Verification solution** for Benzidine, and Benzaldehyde (10ug/mL): 0.5mL of 8270 LCS3 diluted to a final volume of 10ml in Methylene Chloride.

**8.5.5 Continuing Calibration Verification (CCV) solution (10ug/mL):** 0.50mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

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**8.5.6 LCS1/Matrix Spike Spiking Solution (200ug/mL):** Multi component standard commercially available from NSI Environmental Solutions – PN Q3461.

**8.5.7 LCS2/Matrix Spike Spiking Solution (200ug/mL):** this solution is prepared by dilution the following solutions to 10mL in methanol:

**8.5.7.1** 1,2,4,5-tetrachlorobenzene (2000ug/mL): 1.0mL
**8.5.7.2** Benzoic Acid (2000ug/mL): 1.0mL
**8.5.7.3** Pentachloronitrobenzene (5000ug/mL): 0.4mL
**8.5.7.4** 2-Chloroaniline (2000ug/mL): 1.0mL

**8.5.8 LCS3/Matrix Spike Spiking Solution (200ug/mL):** This solution is prepared by diluting the following solutions to 10mL in methanol:

**8.5.8.1** Benzaldehyde Standard (2000ug/mL): 1.0mL
**8.5.8.2** Benzidine Standard (1000ug/mL): 2.0mL

**8.5.9 Internal Standard Primary Standard:** Six component standard commercially available from Restek – PN 31206-510. 2000ug/mL solution containing 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12.

**8.5.9.1 8270 IS working solution (250ug/mL):** 1.25mL of Internal Standard Primary Standard diluted to a final volume of 10mL in methylene chloride. 20uL of the solution is spiked into 1mL sample and standard vials. The final concentration on column is 5.0ug/mL.

**8.5.10 DFTPP Primary Standard:** Four component standard commercially available from UltraScientific – PN GCM-150. 1000ug/mL solution containing Decafluorotriphenylphosphine, Benzidine, Pentachlorophenol,

and 4,4'-DDT.

**8.5.10.1 8270 GC/MS Tuning Solution (50ug/mL):** 1.25mL of DFTPP Primary Standard diluted to a final volume of 25mL in methylene chloride.

# 9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

## 9.1 Blank(s)

**9.1.1 Method Blank:** A method blank must be prepared once per every 20 samples or per extraction batch, whichever is more frequent.

Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Semivolatile Organics Department Manager, Laboratory Director or QA Officer, if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 5x the blank levels. Common laboratory contaminants such as phthalates may be allowed provided that they are at concentration less than five times the reporting limit. In such cases, the sample results are accepted without corrective action and the client is notified in a project narrative associated with the sample results.

## 9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

Laboratory control sample (LCS) and laboratory control duplicate (LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent. The LCS and LCSD are spiked with a solution prepared from a second source or lot number, other than the initial calibration standard used to prepare the curve for the determinative analytical method. The LCS/D may contain all target compounds of interest, or a smaller subset may be analyzed, and is extracted along with the samples as verification of the accuracy of the entire extraction procedure.

Project specific requirements, QAPPs, and/or other DQOs may supersede general limits and QC requirements.

Corrective Action: Analysis must be repeated once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made with approval of the Semivolatile Organics Department Manager, Laboratory Director or QA Officer, if the samples associated with the out of control LCS are also associated with a matrix spike or matrix spike duplicate pair that is in control. This is an acceptable measure of accuracy of the extraction and analytical procedures. In addition, sporadic marginal failure criteria may be employed in some cases. An explanation of this out of control LCS recovery

must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

## 9.3 Initial Calibration Verification (ICV)

The initial calibration for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing a second source calibration standard (Section 8.5.3).

Calculate the percent recovery for each analyte. If the percent recovery for each analyte is within 70-130%, then the calibration is assumed to be valid. Sporadic Marginal Exceedences apply, and in general, "difficult" analytes should be recovered at 40-160% (this is based on MADEP MCP criterion). If this criterion is not met, the standard is reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

## 9.4 Continuing Calibration Verification (CCV)

On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed.

Refer to Section 10.4 for further information.

## 9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples will be performed per batch of 20 samples of the same matrix processed together in those cases where the client does not specify omitting the MS/MSD for the analyses of their samples.

See Section 16 Table E for MS/MSD recovery limits and %RPD limits. Calculate the %RPD as described in Section 9.6.

Corrective Action: Analysis must be repeated once to see if an analytical error has occurred. If the % recovery and/or %RPD still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

## 9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or QAPP request. *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent differences (RPD) of duplicates is listed in Section12. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Analysis must be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD exceedance.

## 9.7 Method-specific Quality Control Samples

**9.7.1 Surrogate Spikes:** Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

See Section 12 for surrogate recovery acceptance criteria.

Corrective Action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

**9.7.2 Internal Standards:** Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes.

The internal standards in the samples should remain at constant area counts with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be 50% to 200% of the Internal Standards in the Continuing Calibration. Additionally, the RRT of the detected sample compounds must be within 0.06RRT units of the retention times of the associated continuing calibration standard.

Corrective Action: Analysis must be repeated once unless there are obvious samples matrix interferences, e.g. If the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-analysis.

## 9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

# 10. Procedure

## 10.1 Equipment Set-up

**10.1.1** The **basic GC parameters** are as follows:

Injection Port Temp: 260°C
Oven Equib Time: 0.50 min
Oven Max: 350°C
Oven: On
Cryo: Off
Ambient: 45°C
Cryo Blast: Off

Initial Temp.: 42°C
Initial Time: 2.00 min

| Level | Rate (°C /min) | Final Temp. (°C) | Final Time (min) |
|---|---|---|---|
| 1 | 25.00 | 250 | 0 |
| 2 | 10.00 | 320 | 2 |
| | | | 19.32 |

Final Time: 19.32
Please note that this may differ slightly from one instrument to another.

The **basic injection port parameters** are as follows:
Pulsed Splitless "Split" mode
Equilibrium Time: 0.50 minute
Temp: 260°C
Pressure: 9.2752 psi
Total Flow: 34.2mL/min
Split: NA
Split Flow: NA
Septum Purge Flow: 3mL/min
Injection Pulse Pressure: 30psi Until 0.2min
Purge Flow to Split Vent: 30mL/min at 0.6 min
Injection volume: 1uL

Please note that this may differ slightly from one instrument to another.

**10.1.2** **Column conditions** parameters are as follows:

Constant Pressure: 9.2752 psi
Column flow: 1.2mL/min
Linear Velocity: 39.763 cm/sec

Please note that this may differ slightly from one instrument to another.

**10.1.3** **Mass Spectrometer** Scan Parameters
Scan from mass 35.0 amu to 450 amu

### 10.1.4 Tuning

Tune acceptance should be verified at the beginning of every 12 hour analytical shift. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met.

Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1 uL of a 50 ug/mL DFTPP solution.

Note: Within the Enviroquant software, Autofind is used first to evaluate Tune. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

Average or single scan may be used, if Autofind does not pass, but caution should be used in tuning on a single scan too close to the front or back of the DFTPP peak. If the system will not tune under ideal conditions, injection port maintenance and/or a source clean should be performed.

The response of Benzidine and Pentachlorophenol, when monitored on a daily basis, will indicate the efficiency of the chromatography system. Benzidine and Pentachlorophenol should be present at their normal responses and no peak tailing should be visible. The tailing factor of Benzidine and Pentachlorophenol must not exceed 2. DDT breakdown must be <20%. Although moderate tailing may indicate maintenance on the instrument will be required soon, these additional compounds in the DFTPP standard are included as an aid in instrument performance evaluation.

The following DFTPP mass intensity criteria should be used:

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

| Mass m/z | Abundance criteria |
|---|---|
| 51 | 10-80% of Base Peak. |
| 68 | Less than 2% of mass 69. |
| 70 | Less than 2% of mass 69. |
| 127 | 10-80% of Base Peak. |
| 197 | Less than 2%of mass 198. |
| 198 | Base peak, or > 50% of Mass 442. |
| 199 | 5-9% of mass 198. |
| 275 | 10-60% of Base Peak. |
| 365 | Greater than 1% of mass 198. |
| 441 | Present but less than 24% of mass 442. |
| 442 | Base Peak, or > 50% of mass 198. |
| 443 | 15-24% of mass 442. |

## 10.2 Initial Calibration

**10.2.1** After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.5.3.3 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in the standards freezer. When the Initial Calibration is typed into a sequence, the standard ID for each of the levels must be noted in the "comments" of the sequence. (Refer to the Reagent Solvent and Standard Control SOP (G-008) for further information regarding standard labeling convention.)

**10.2.2** Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

The RSD of each target analyte, for each level in the ICAL, must meet the minimum response factor criteria found in Table C. The %RSD for each target analyte must not exceed 20%. If the %RSD for any target exceeds 20%, then the linear calibration model must be employed. A linear fit is acceptable if the correlation coefficient is greater than 0.99. When calculating calibration curves using the linear regression model, a minimum quatitiation check must be performed on the lowest calibration point. The recalculated concentration of the lowest calibration point should be within 30% of the true value. If this criteria is not met then the RL must be raised to the concentration of the next calibration standard that exhibits acceptable recoveries when recalculating using the final calibration curve. Up to 10% of the target analytes may have a %RSD greater than 20%, or a correlation coefficient less than 0.99, but these compounds must be reported as estimated values.

**10.2.3** Once the calibration curve is accepted, the Initial Calibration Verification (ICV) must be analyzed.

Reference standards from a separate source or different lot are analyzed after every initial calibration for evaluation against calibration standard solutions. The percent difference shall not be greater than 30% for all target compounds. The response factors for all compounds must meet the minimum RF listed in table C. Allowances are made for limited %D failures for the number of compounds analyzed. Through historical data Benzaldehyde, Benzoic Acid, and Benzidine are problematic compounds and are not included within the Tolerated Failures listed below. Based on the current literature, Benzidine and Benzaldehyde are subject to oxidative losses and exhibit erratic and non-reproducible chromatographic behavior. As such, these compounds are evaluated only when client specified based on the following: a %D exhibiting a low bias should not be below 40%D, however in a high bias situation the %D may be greater than 40%. If associated samples are non-detect for these compounds and the %D is a high bias, no corrective action is needed and the ICV and/or CCV can be accepted. Alpha Analytical – Mansfield Lab reports these compounds by default unless the client specifies they do not require them.

| Number of Target Analytes | Number of Tolerated Failures |
|---|---|
| 11-30 | 1 |
| 31-50 | 2 |
| 51-70 | 3 |
| 71-90 | 4 |
| >90 | 5 |

## 10.3 Equipment Operation and Sample Processing

### 10.3.1 Sample Extraction and Cleanup

Samples for 8270 are generally extracted using one of the following SOPs:

- *Shaker Table Extraction* (OP-013)
- *Separatory Funnel Extraction* 3510C (OP-001)
- *Microscale Solvent Extraction* 3570 (OP-016)

Samples for 8270 may be cleaned up before transfer to the instrument laboratory using one of the following SOPs:

- *Gel Permeation Column Cleanup* SOP (OP-006)
- *Silica Gel Clean-up* SOP (OP-014) – limited target compounds only.

**10.3.2** The preparation lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 1.0 to 5.0mL final volume; 1.0mL is transferred and any remaining sample is put into archive. One aliquot of each sample is then logged into the Internal Chain of Custody book and placed in the sample extract holding refrigerator located in the instrument laboratory.

**10.3.3** All samples and standards are spiked with the six Internal Standards (IS) before analysis, Section 8.5.9.1. The internal standard is intended to be used for both quantitation and the establishment of relative retention times.

**10.3.4** The analyst may determine to screen any samples for needed dilution. A sample that will need a dilution either for target analytes that are over calibration, or samples may need to be diluted solely for matrix issues when the GC/MS chromatogram shows a large UCM or other non-target interferences, or when the extract itself is very colored and viscous. All of the samples at 1mL (including the QC samples of a method blank and LCS) are spiked with 20uL of internal standard for a concentration of 5ug/mL. The samples are shaken briefly after the internal standard is added to ensure mixing.

**10.3.5** After the samples have been analyzed, the data files from the 5973 MS are transferred to the server. The samples are quantitated versus the proper method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria. The istdrpt macro may, also, be utilized to check the internal standard and surrogate criteria. The following should be reviewed initially:

**10.3.5.1** Are all the surrogates within QC criteria? Please see Section 12 for surrogate information.

**10.3.5.2** Are all the internal standards within 50-200% and within ±0.06 RRT of the daily CCAL? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

**10.3.5.3** Are all target analytes within calibration range? If not, the sample should be diluted and re-analyzed. Samples may still be outside calibration range even though they were screened and it appeared that they did not need a dilution, or the sample may need additional dilution to put target analytes within calibration range. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to provide a concentration of 5ug/mL. Conversely, if a sample has been over-diluted, it may need to be analyzed at less of a dilution to detect target analytes that may have been diluted out.

**10.3.5.4** Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it will need to be re-analyzed in another tune clock.

**10.3.6** The sequence should also be printed out from Chemstation, initialed and dated, placed in the working logbook.

**10.3.7** If anything in the initial review of the data indicates that there should be a re-analysis or a re-extract, the reason for re-analysis or re-extract should be noted on the sequence.

**10.3.8** If a re-extract is required, the “Request for Reanalysis/Repreparation” book should be filled out and a photocopy of the appropriate page should be given to the Preparation Group leader or the Semivolatile Organics Department Manager.

**10.3.9** A standard sequence (saved as S8073001;”S” for semivolatile;”8” for BNA8; 0730 for July 30, and 01 is the first sequence that day) would appear as follows:

Tune: WG#-1, -4, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the Tune.

CCV: WG#-2, -3, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the calibration verification.

## 10.4 Continuing Calibration Verification (CCV)

**10.4.1** On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are:

**10.4.1.1** The RFs observed for all targets must mee the minimum RFs listed in Table C. The %D for all targets must be ≤20%. Up to 20% of all targets are allowed to be > 20%. All target compounds exhibiting a %D greater than 20% must be reported as estimated if present. ND results for compounds in which the CCV exhibits a high bias are acceptable. The retention times of the internal standards must be within 0.5min of the mid-level in the previous ICAL.

**10.4.1.2** The area counts of the internal standards must be within 50 to 200% of the mid-level in previous ICAL.

### 10.5 Preventative Maintenance

All repair and non-routine maintenance records including outside service visits are maintained in the instrument maintenance logbooks.

**Injection Port Maintenance:** Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed.

**Septum Maintenance:** The septum needs to be changed approximately every two-hundred injections. Unscrew the top septum nut, remove the pierced septum, and replace with a new 11mm Thermolite green septum (Restek) or equivalent. Screw the top septum nut back on.

**Column Maintenance:** Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner, and the inlet nut is tightened.

## 11. Data Evaluation, Calculations and Reporting

### 11.1 Qualitative Analysis

**11.1.1** The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

**11.1.2** The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting this criteria.

**11.1.3** The relative retention time of the sample component is within $\pm$ 0.06 RRT units of the RRT of the standard component, taking into consideration matrix effects which may cause retention time shifts.

**11.1.4** The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Outlying abundances maybe included due to the presence of non-target interference.

**11.1.5** Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

**11.1.6** Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

**11.1.7** Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute, (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum may contain extraneous ions contributed by the coeluting compound.

**11.1.8** For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification (TICs) will be determined by the purpose of the analyses being conducted. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**11.1.8.1** For example, the RCRA permit or waste delisting requirements may require the reporting of non target analytes. Only after a visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

**11.1.8.1.1** Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

**11.1.8.1.2** The relative intensities of the major ions should agree within ± 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).

**11.1.8.1.3** Molecular ions in the reference spectrum should be present in the sample spectrum.

**11.1.8.1.4** Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or presence of co-eluting compounds.

**11.1.8.1.5** 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 peaks. Data system library reduction programs can sometimes create these discrepancies.

## 11.2 Quantitative Analysis

**11.2.1** Response factors and % RSD to evaluate Initial Calibration acceptability.

*Calculate RF by:*

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

*area cmp* = Area of the characteristic ion for the compound being measured.
*area is* = Area of the characteristic ion for the specific internal standard.
*conc. is* = Concentration of the specific internal standard.
*conc. cmp* = Concentration of the compound being measured.

*Calculate %RSD by:*

$$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}}$$

where:

*%RSD* = Percent Relative Standard Deviation
*x* = Average of RF's
*SD* = Standard Deviation
$x_i$ = Analytical results of each level in the final reporting units
*N*=Number of results (levels)

**11.2.2** For compounds in the initial calibration in which the Average of Response Factors calibration model is used:

*Calculate %Difference (%D):*

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi - Initial Calibration average RF
RFc = Continuing Calibration RF

**11.2.3** For compounds in the Initial Calibration for which the Linear Regression calibration model is used:

$$\% \text{ Drift} = \frac{\text{Calculated [ ] – Theoretical [ ]}}{\text{Theoretical [ ]}} \times 100$$

**11.2.4** Results of Water Analysis- calculation as performed in report form:

$$\text{Concentration (ug/L)} = \frac{\text{(Conc) (Vf) (DF)}}{\text{(Vi)}} \times 1000$$

where:
Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
Vf = Final volume of extract (mL)

Vi = Volume of sample extracted (mL)
DF = Dilution factor, for manually prepared dilutions

**11.2.5** Results of Sediment/Soil and Sludge Analysis- calculation as performed in report form:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc})\,(\text{Vf})\,(\text{DF})}{(\text{W})\,(\%\text{S})} \times 1000$$

where:

Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Vf = Extract final volume (mL)
W = Aliquot of sample (wet), g
%S = Sample % solid (in decimal form)

# 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All results for the organic compounds of interests are reportable without qualification if extraction and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

If non-compliant organic compound results are to be reported, the Semivolatile Organics Department Manager, the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

| QC Parameter | Acceptance Criteria |
|---|---|
| Initial Calibration Curve | ≤20%RSD or ≥0.99 linear for all targets. Up to 10% of compounds can fail to meet this criteria, but must be reported as estimated. If linear regession is used, recalculation of the lowest calibration standard is required to be within 30% of the true value. All RFs must meet the minimum RFs listed in Table C. |
| Independent Check Verification | +/- 30%D for all compounds<br>Up to 5 sporadic marginal failure accepted |
| Continuing Calibration Verification | Analyzed every 12hr or at the minimum of every 20 samples;<br>≤ 20%D for all compounds. Minumum RFs listed in Table C must be met.<br>Up to 5 sporadic marginal failure criteria accepted |
| Method Blank | No analyte detected at or above the reporting limit,<br>"B" qualify analyte if detected concentration is less than 5X the concentration found in the associated sample(s) |

| | |
|---|---|
| Laboratory Control Sample And LCSD | 40-140% (Base/Neutral compounds) and 30-130% (Acid compounds) for all matrices; target specific<br>30% RPD |
| Matrix Spike / Matrix Spike Duplicate | Same as for LCS/D; 30% RPD between the duplicates. |
| Sample / Sample Duplicate | 30% RPD between the duplicates. |
| Surrogates | Soil: 30-130%<br>Waters: 30-130% for BN and 15-115% for Acid |
| Internal Standards | 50% - 200% of the daily CCV area for the Internal Standards |
| Project specific QAPP and DQOs supersede the above limits. | |

# 13. Method Performance

## 13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

## 13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

### 13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

### 13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

# 14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOPs for further pollution prevention and waste management information.

# 15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

Waste Management and Disposal SOP

SOP/08-03 Manual Integration

# 16. Attachments

***Table A: Surrogate Recovery Acceptance Criteria***

Water: 15-115% for Acid compounds and 30-130% for Base/Neutral Compounds
Soils: 30-130%

Note: The above limits will be replaced by laboratory limits, when available. Limits are determined by sample DQO and matrix. The limits above may also vary with project specific requirements, QAPP, or DQOs.

***Table B: Target Compounds and Quantitation Ions***

| Compound | Quantitation Ions Primary | Secondary |
|---|---|---|
| Aniline* | 93 | 39, 93 |
| Acenaphthene (CCC) | 153 | 154, 152 |
| Acenaphthene-d10 (IS) | 164 | 162, 160 |
| Acenaphthylene | 152 | 151, 153 |
| Acetophenone | 105 | 77, 51 |
| Anthracene | 178 | 176, 179 |
| Azobenzene * | 182 | 77 |
| Atrazine | 200 | 173, 215 |
| Benzaldehyde | 77 | 105, 106 |
| Benzo(a)Anthracene | 228 | 229, 226 |
| Benzo(a)pyrene D12 (SURR) | 264 | 132 |
| Benzidine* | 184 | 183,92 |
| Benzo(b)fluoranthene | 252 | 253, 125 |
| Benzo(k)fluoranthene | 252 | 253, 125 |
| Benzo(g,h,i)perylene | 276 | 138, 277 |
| Benzo(a)pyrene | 252 | 253, 125 |
| Benzoic Acid* | 105 | 122, 77 |
| Biphenyl | 154 | 153, 76 |
| Benzyl Alcohol | 108 | 79, 77 |
| Bis(2-chloroethoxy)methane | 93 | 95, 123 |
| Bis(2-chloroethyl)ether | 93 | 63, 95 |
| Bis(2-chloroisopropyl)ether | 45 | 77, 79 |
| Bis(2-ethylhexyl)phthalate | 149 | 167, 279 |
| 4-Bromophenylphenylether | 248 | 250, 141 |
| Butylbenzylphthalate | 149 | 91, 206 |
| Carbazole* | 167 | 139,166 |
| Caprolactam | 113 | 55, 56 |
| 2-Chloroaniline | 127 | 129 |
| 4-Chloroaniline | 127 | 129 |
| 4-Chloro-3-methylphenol (CCC) | 107 | 144, 142 |
| 2-Chloronaphthalene | 162 | 127, 164 |
| 2-Chlorophenol | 128 | 64, 130 |
| 4-Chlorophenylphenylether | 204 | 206, 141 |
| Hexachlorobenzene | 284 | 142, 249 |
| Chrysene | 228 | 226, 229 |
| Chrysene-d12 (IS) | 240 | 120, 236 |

***Table B, continued: Target Compounds and Quantitation Ions***

| Compound | Quantitation Ions Primary | Secondary |
|---|---|---|
| Dibenz(a,h)anthracene | 278 | 139, 279 |
| Dibenzofuran | 168 | 139 |
| Di-n-butylphthalate | 149 | 150, 104 |
| 1,2-Dichlorobenzene | 146 | 148, 113 |
| 1,3-Dichlorobenzene | 146 | 148, 113 |
| 1,4-Dichlorobenzene (CCC) | 146 | 148, 113 |
| 1,4-Dichlorobenzene-d4 (IS) | 152 | 115 |
| 3,3'-Dichlorobenzidine* | 252 | 254, 126 |
| 2,4-Dichlorophenol (CCC) | 162 | 164, 98 |
| Diethylphthalate | 149 | 177, 150 |
| 2,4-Dimethylphenol | 107 | 122, 121 |
| Dimethylphthalate | 163 | 194, 164 |
| 4,6-Dinitro-2-methylphenol | 198 | 182,77 |
| 2,4-Dinitrophenol (SPCC) | 184 | 63, 154 |
| 2,4-Dinitrotoluene | 165 | 63, 182 |
| 2,6-Dinitrotoluene | 165 | 63, 121 |
| Di-n-octylphthalate (CCC) | 149 | 167, 150 |
| Fluoranthene (CCC) | 202 | 101, 100 |
| Fluoranthene D10 (SURR) | 212 | 106 |
| Fluorene | 166 | 165, 167 |
| 2-Fluorobiphenyl (SURR) | 172 | 171,173 |
| 2-Fluorophenol (SURR) | 112 | 64,91 |
| Hexachlorobutadiene (CCC) | 225 | 223, 227 |
| Hexachlorocyclopentadiene (SPCC) | 237 | 235, 272 |
| Hexachloroethane | 117 | 201, 199 |
| Isophorone | 82 | 95, 138 |
| Indeno(1,2,3-cd)pyrene | 276 | 138, 227 |
| 2-Methylnaphthalene | 142 | 141 |
| 2-Methylphenol | 108 | 107 |
| 4-Methylphenol | 108 | 107 |
| Naphthalene | 128 | 129, 127 |
| Naphthalene-d8 (IS) | 136 | 68 |
| 2-Nitroaniline | 65 | 92, 138 |
| 3-Nitroaniline | 138 | 108, 92 |
| 4-Nitroaniline | 138 | 108, 92 |
| Nitrobenzene | 77 | 123, 65 |
| Nitrobenzene-d5 (SURR) | 82 | 128, 54 |
| 2-Nitrophenol (CCC) | 139 | 109, 65 |
| 4-Nitrophenol (SPCC) | 109 | 139, 65 |
| N-Nitrosodiphenylamine (CCC) | 69 | 168, 167 |
| N-Nitroso-di-n-propylamine (SPCC) | 169 | 42, 101, 130 |
| N-Nitrosodimethylamine* | 74 | 42, 44 |
| Pentachlorophenol (CCC) | 266 | 264, 268 |
| Pentachloronitrobenzene | 237 | 214, 249 |
| Perylene-d12 (IS) | 264 | 260, 265 |
| Phenanthrene | 178 | 179, 176 |
| Phenanthrene-d10 (IS) | 188 | 94, 80 |
| Phenol (CCC) | 94 | 65, 66 |

***Table B, continued: Target Compounds and Quantitation Ions***

| Compound | Quantitation Ions Primary | Secondary |
|---|---|---|
| Phenol-d5 (SURR) | 99 | 42,71 |
| Pyrene | 202 | 200, 203 |
| Pyridine | 79 | 52 |
| Terphenyl-d14 (SURR) | 244 | 122. 212 |
| 1,2,4,5-Tetrachlorobenzene | 216 | 214, 179 |
| 2,3,4,6-Tetrachlorophenol | 232 | 131, 230 |
| 2,4,6-Tribromophenol (SURR) | 330 | 332, 141 |
| 1,2,4-Trichlorobenzene | 180 | 182, 145 |
| 2,4,5-Trichlorophenol | 196 | 198, 97, 132, 99 |
| 2,4,6-Trichlorophenol (CCC) | 196 | 198, 200 |

* Indicates compound is not part of standard reporting list.
**Note:** Target list will vary based on project specific requirements.

***Table C: Recommended Minimum Response Factor Initial and Continuing Calibration***

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| Benzaldehyde | 0.01 |
| Phenol | 0.8 |
| Bis(2-chloroethyl)ether | 0.7 |
| 2-Chlorophenol | 0.8 |
| 2-Methylphenol | 0.7 |
| 2,2'-Oxybis-(1-chloropropane) | 0.01 |
| Acetophenone | 0.01 |
| 4-Methylphenol | 0.6 |
| N-Nitroso-di-n-propylamine | 0.5 |
| Hexachloroethane | 0.3 |
| Nitrobenzene | 0.2 |
| Isophorone | 0.4 |
| 2-Nitrophenol | 0.1 |
| 2,4-Dimethylphenol | 0.2 |
| Bis(2-chloroethoxy)methane | 0.3 |
| 2,4-Dichlorophenol | 0.2 |
| Naphthalene | 0.7 |
| 4-Chloroaniline | 0.01 |
| Hexachlorobutadiene | 0.01 |
| Caprolactam | 0.01 |

***Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration***

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| Hexachlorocyclopentadiene | 0.05 |
| 1,1'-Biphenyl | 0.01 |
| 2-Chloronaphthalene | 0.8 |
| 2-Nitroaniline | 0.01 |
| Dimethyl phthalate | 0.01 |
| 2,6-Dinitrotoluene | 0.2 |
| Acenaphthylene | 0.9 |
| 3-Nitroaniline | 0.01 |
| Acenaphthene | 0.9 |
| 2,4-Dinitrophenol | 0.01 |
| 4-Nitrophenol | 0.01 |
| Dibenzofuran | 0.8 |
| 2,4-Dinitrotoluene | 0.2 |
| Diethyl phthalate | 0.01 |
| 1,2,4,5-Tetrachlorobenzene | 0.01 |
| 4-Chlorophenyl-phenyl ether | 0.4 |
| Fluorene | 0.9 |
| 4-Nitroaniline | 0.01 |
| 4,6-Dinitro-2-methylphenol | 0.01 |
| 4-Bromophenyl-phenyl ether | 0.1 |
| N-Nitrosodiphenylamine | 0.01 |
| Hexachlorobenzene | 0.1 |
| Atrazine | 0.01 |
| Pentachlorophenol | 0.05 |
| Phenanthrene | 0.7 |
| Anthracene | 0.7 |
| Carbazole | 0.01 |
| Di-n-butyl phthalate | 0.01 |
| Fluoranthene | 0.6 |
| Pyrene | 0.6 |
| Butyl benzyl phthalate | 0.01 |
| 3,3'-Dichlorobenzidine | 0.01 |
| Benzo(a)anthracene | 0.8 |
| Chrysene | 0.7 |
| Bis-(2-ethylhexyl)phthalate | 0.01 |
| Di-n-octyl phthalate | 0.01 |

***Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration***

| Semivolatile Compounds | Minimum Response Factor (RF) |
|---|---|
| Benzo(b)fluoranthene | 0.7 |
| Indeno(1,2,3-cd)pyrene | 0.5 |
| Dibenz(a,h)anthracene | 0.4 |
| Benzo(g,h,i)perylene | 0.5 |
| 2,3,4,6-Tetrachlorophenol | 0.01 |