METHOD 8270 ANALYSIS OF
SEMIVOLATILE ORGANIC COMPOUNDS BY
COMBINED GAS CHROMATOGRAPHY/ MASS
SPECTROMETRY (GC/MS)

REV 7

Copy No. _______

Revised By: 
Date: 3/16/2011

Technical Review: 
Date: 3/17/2011

QA Review: 
Date: 3/18/11

Director: 
Date: 3/17/2011
1.0 SCOPE

Method 8270 is a combined Gas Chromatography/Mass Spectrometry (GC/MS) method used for the identification and quantitation of a wide range of organic compounds and is suitable for extracts generated from soil, water, air, biota, or chemical waste media. Table 1 of the US EPA method 8270D is a comprehensive listing of compounds amenable for identification using this methodology. The laboratory however, in the absence of a client-specified target analyte list (TAL), will analyze for the compounds listed in Table 11 of this TAP.

Estimated quantitation limits for samples prepared as described in the method are 330 to 1000 µg/kg for soil/sediment, 10 to 50 µg/L for water, and 1.0 to 200 mg/kg for wastes. Quantitation limits are highly dependent on the sample matrix.

The following compounds may require special attention when being determined by this method:

benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, N-nitrosodiphenylamine, 1,2-diphenylhydrazine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-nitroaniline, benzyl alcohol, and benzoic acid

This method is not suitable for the quantitation of multi-component analytes such as Toxaphene, Aroclors, and Technical Chlordane due to low sensitivity for these analytes in the electron impact (EI) mode of ionization. It can, however, be used as a confirmatory method if the concentrations in the extracts generated for Methods 8081 and 8082 are sufficiently high enough to be detected by GC/MS.

Modifications to this method may be performed but should be performed with the prior approval of the client and should be documented in the case narrative of a data package.

2.0 PURPOSE

To describe the procedure for the analysis of samples for semi-volatile compounds by combined gas chromatography/mass spectrometry (GC/MS) by Method 8270D.
3.0  RESPONSIBILITIES

3.1  It is the responsibility of the Study Director (Project Manager) to assure that all steps described in this procedure are performed.

3.2  It is the responsibility all laboratory personnel to follow the procedure.

4.0  REFERENCES

4.1  Identification of the Test Method(s):

**SW-846 Methods:**

8270C, Revision 3, December 1996
8270D, Revision 4, February 2007
8000C, Revision 3, March 2003

4.2  For extraction, refer to the following SwRI Test/Analytical Procedures:

4.2.1  **TAP-01-0402-019**, Extraction of Base/Neutral and Acid Semivolatiles from Water by Separatory Funnel by Method 8270

4.2.2  **TAP-01-0402-113**, Extraction of Base Neutral and Acid Semivolatiles (BNAs, 8270C) from Water by Continuous Liquid – Liquid Extraction

4.2.3  **TAP-01-0402-020**, Extraction by Sonication of Base Neutral and Acid Semivolatiles (BNAs), Pesticides and/or PCBs (Aroclors) from Soils/Sediment for GC and GC/MS Analysis by Methods 8081, 8082, and 8270C.

4.2.4  **TAP-01-0402-099**, Preparation and Extraction of Soil and/or Sediment Matrices Samples by Soxhlet for 8270C BNA Analysis

4.3  Reference/Operations manuals for instruments utilized are readily available for use in troubleshooting and maintenance of equipment.
5.0 PROCEDURE

5.1 Definitions

5.1.1 Contract Required Detection Limit (CRQL) – The detection limit, usually in parts per billion (ppb), is outlined in the contractual agreement between the client and the provider of analytical services. This is not the Method Detection Limit.

5.1.2 Method Detection Limit (MDL) – The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero, as determined from analysis of a sample containing the analyte in a given matrix.

5.1.3 The laboratory will also establish a Level of Detection (LOD) for each analyte of interest. This LOD may not necessarily be the same value as the MDL.

5.1.4 Definitions are provided in QAP-004, Quality Assurance Plan for Analytical and Environmental Services.

5.2 Level of Quantitation (LOQ)

The LOQ is typically near the lab’s low point calibration standard and will be established for each analyte. In all cases, the LOQ is higher than the LOD. In SIM mode, lower LOQs can be achieved.

5.3 Interferences

5.3.1 Raw GC/MS data from all Blanks, samples and spikes shall be evaluated for interferences. The source of interference shall be determined whether it is in the preparation and/or clean up of the samples. Corrective action should be taken to eliminate the problem.

5.3.2 Occasional carryover contamination may occur whenever high concentration samples are analyzed. To reduce carryover contamination,
an instrument blank shall be analyzed especially when dirty samples are analyzed.

5.4 Equipment and Supplies

5.4.1 Gas Chromatograph/Mass Spectrometer system

5.4.1.1 Gas Chromatograph – A Agilent 6890N Network GC equipped with a Agilent 7683B auto-sampler, split/splitless injection, and temperature programmable capabilities with other accessories, such as syringes, gas lines, etc.

(a) Alternate GC - A Agilent 7890A GC system equipped with a 7683 injector auto sampler, split/splitless injection and temperature programmable capabilities with other accessories, such as syringes, gas lines, etc.

5.4.1.2 Analytical Column:

(a) A fused silica capillary column 30m long with a 0.25mm ID [or 0.18mm ID if needed] 0.25µm film thickness [J&W Scientific DB-5MS or equivalent].

(b) A capillary column 20m long with 0.18mm ID and 0.36µm film thickness [J&W Scientific DB-5.625 or equivalent].

5.4.1.3 Mass Spectrometer – 1.) Agilent 5975 Inert MSD capable of scanning from 35 to 550amu every 1 second or less, using 70 volts nominal electron energy in the electron impact ionization mode. 2.) Agilent 5975C Inert MSD with triple axis detector capable of scanning from 35 to 550amu every 1 second or less, using 70 volts nominal electron energy in the electron impact ionization mode.

5.4.1.4 GC/MS Data System – Data acquisition and reduction is accomplished by using a PC interfaced with MS ChemStation.
Software. Data processing software is capable of plotting Extracted Ion Current Profile [EICP] which is a plot of ion abundances versus time.

5.4.1.5 Accessories

(a) Syringes of various sizes capable of measuring standards from 5µL to 1mL.

(b) Volumetric flasks – Appropriate sizes with ground-glass stoppers.

(c) Analytical balance – Capable of weighing 0.0001g.

(d) Glass vials – Various sizes to hold standard stock solutions.

5.5 Reagents and Standards

5.5.1 Preservation, Shipment, and Storage of Standards

5.5.1.1 The following are the general procedures to be followed for correct usage, storage, and inventory of chemicals used in the GC/MS labs for semivolatile analysis.

5.5.1.2 Ordering of New Chemicals - No new chemicals are to be ordered until a thorough check has been completed to ensure that the chemical is not available in-house. If a chemical must be purchased, efforts must be made to obtain the minimum amount required. Material Safety Data Sheets and Certificates of Analysis must be explicitly requested on the purchase requisition when a chemical is ordered.

5.5.2 Receipt and Login

Chemicals must be logged into the appropriate freezer. Their receipt must be documented in the database used for chemical inventory. Date received
must be marked on the bottle. This database should contain lot number, date received, amount received, type of usage expected (ie, Appendix IX, Met 8270, etc), and the chemical's expiration date. The MSDS sheet must be filed in alphabetical order using the chemical's most common name.

5.5.3 Usage

When the container is first opened, date opened must be clearly marked on the outside of the bottle.

5.5.4 Expiration Dates and Chemical Disposal:

Except in special circumstances, such as with chemicals that are extremely volatile or unstable, neat chemicals will be stored for an indefinite period of time. Due to the high costs of disposal and the need to stock a wide variety of chemicals required for preparation of standards, it is, in general, more cost efficient to retain chemicals and evaluate their suitability each time their use is required. Since the chemicals used for semi-volatile analysis are often required on a sporadic basis for qualitative standards, it is possible to evaluate them for degradation prior to each use by preparing a stock solution and analyzing it by GC/MS.

Under certain circumstances such as those outlined below, chemicals must be removed from inventory and properly disposed of if:

- The label has peeled off or is no longer legible and the identity of the compound cannot be conclusively determined, a stock solution of the chemical must be prepared and analyzed by GC/MS. Once a tentative identification is made, the chemical must be disposed of according to procedures established by the Safety Office.

- Chemicals showing obvious signs of degradation or decomposition, such as rusting around the lid or marked discoloration, crusting, or abnormal sedimentation, must also be removed from inventory and disposed of properly.
When a given chemical is used to prepare a standard, the entry in the standards-preparation log-book must contain the following information, if available, vendor or supplier, percent purity, lot number, date prepared, and name of analyst preparing the solution.

In all cases, when a chemical is used to prepare a standard, the suitability of available inventory of the given chemical must be evaluated for the specific application required, i.e. an out-dated chemical will be appropriate for preparing a qualitative standard in which precise quantitation is not necessary, but should not be used to prepare a standard used in a project requiring rigorous quality control and precise quantitation of the analytical standard.

5.5.5 All standards and solutions shall be recorded in a standards notebook and appropriately labeled.

5.5.6 Standards used in this laboratory may be purchased as mixes or as individual components from various commercial sources. These standards may then be diluted to the working level or combined with other mixes to make the following:

- INTERNAL STANDARD solution
- DFTPP solution
- MATRIX SPIKE solution
- SURROGATE solution
- INITIAL and CONTINUING CALIBRATION standards.

**Note:** All solutions should be put back into the appropriate freezer as soon after injection onto the GC/MS system as possible in order to preserve the integrity of the solutions. Stock standard solutions and working level solutions will be prepared at a minimum of every 12 months. If degradations are detected, standards will be prepared on a more frequent basis.

5.5.7 Before beginning preparation of the above-mentioned solutions, make sure that the steps mentioned below are followed:
5.5.7.1 Allow mixes to come up to room temperature.

5.5.7.2 Check mix for any precipitates - if any, the mix may be warmed up using hot tap water to get precipitates back into solution.

5.5.7.3 Additionally, a sonicator may be used in conjunction with the warm tap water to get precipitates back into solution.

5.5.7.4 Check all syringes and volumetric glassware for cleanliness and dryness.

5.5.7.5 Carefully transfer mixes from ampoule to clean vials.

5.5.8 **Tuning Check Solution** containing Decafluorotriphenylphosphine [DFTPP], Pentachlorophenol, Benzidine, and 4,4'-DDT @ 50 or 25 ng/µL.

5.5.9 **Semi-volatile Internal Standard Solution** containing six internal standards is prepared at 1000 ng/µL.

5.5.10 Additional notes:

5.5.10.1 Internal Standard is spiked into every sample analyzed prior to injection onto the GC/MS system.

5.5.10.2 To a 200uL aliquot of a water or soil sample extract, add 4 µL of internal standard solution (at 1000 ng/µL) such that the concentration of the internal standards is at 20 ng/µL.

5.5.11 **Semi-volatile Surrogate Spiking Solution** is prepared at 100 µg/mL for Base/Neutral components and 150 µg/mL for Acid components.

**Note:** The surrogate spiking solution, once prepared, should be given to the extraction laboratory.
5.5.12 **Semi-volatile Matrix Spike Spiking Solution** is prepared at 100 µg/mL for spiked components from second source different with one for initial calibration standards.

**Note:** The matrix spike solution, once prepared, should be given to the extraction laboratory.

5.5.13 A stock solution containing all target analytes is prepared and dilutions can be made to prepare the **Initial and Continuing Calibration** solutions.

### 5.6 Quality Control

#### 5.6.1 Laboratory Method Blank

Analysis of a laboratory method blank serves the purpose of checking the purity of solvents and reagents used in the preparation/extraction of a set of samples as well as provide information regarding potential sources of contamination.

No interferences can be present which may affect the identification of target analytes. Detected analytes shall be less than the Contract Required Quantitation Limit [CRQL]. Phthalate esters detected shall be less than 2.5 times the CRQL.

#### 5.6.2 Surrogate Recoveries

All samples, blanks, and MS/MSD will be checked for surrogate recoveries. The QC limits are as stated in Table 2 or from updated control charts. If surrogate recoveries are not met, the following corrective action must be taken:

- Check instrument for malfunction.
- Check calculations (i.e.-surrogate, internal standard, and raw data).
- Re-analyze sample extract, as per Project Manager or client request.
If upon reanalysis, the problem still exists, sample re-extraction and re-analysis may be required upon client request. Otherwise, the data shall be flagged “estimated concentration”.

5.6.3 Matrix Spike Recoveries. The QC limits are as stated in Table 3 or updated control charts. For MS/MSDs outside criteria, corrective action may only be required if LCS recoveries also fall outside QC limits. If calibration method blank, and LCS meet criteria, these MS/MSD recovery criteria may be attributed to matrix effects.

5.6.3.1 An MS/MSD shall be extracted at a frequency of one pair per extraction batch of 20 samples or less per SDG unless otherwise instructed by the Project Manager or the client.

5.6.4 In addition to an MS/MSD pair, a Laboratory Controlled Sample [LCS] shall be extracted at a frequency of one per extraction batch of 20 samples or less per SDG per sample matrix unless otherwise instructed by the Project Manager or the client. LCS recoveries shall meet QC limits as shown in Table 3 or updated control charts.

Note: For projects that require NELAC protocol to be followed, at a minimum, one set of sample MS/MSD, and one LCS sample MUST be generated per extraction batch of 20 samples or less per SDG per sample matrix without exception.

5.6.5 For compounds spiked into the LCS, MS, and MSD other than those listed in Table 3, the advisory limits shall be 20-150%.

5.6.6 Initial Demonstration of Proficiency (IPR)

The laboratory shall generate accuracy and precision data to demonstrate proficiency in sample preparation and analysis as per section 8.0 of Method 8000C.

5.6.7 In-house QC limits
5.6.7.1 In-house QC limits for recovery of matrix spike, LCS, IPR, and OPR shall be generated using a minimum of 15-20 data points.

5.6.7.2 As per section 9.7.3 of Method 8000C, control limits and warning limits shall be calculated in the following manner:

Upper Control Limit (UCL) = X + 3S
Lower Control Limit (LCL) = X - 3S

Upper Warning Limit (UWL) = X + 2S
Lower Warning Limit (LWL) = X - 2S

Where
X is the mean recovery
S is the standard deviation

5.6.8 50ng on column of DFTPP (Decafluorotriphenylphosphine) are injected and a mass listing is generated. Relative abundances should meet the criteria in Table 1 for a mass spectrum generated by MSD.

Alternatively, other documented tuning criteria may be used (e.g. CLP, or Method 625), provided that method performance is not adversely affected and used consistently throughout the initial calibration, calibration verification, and sample analyses.

5.6.9 If these criteria are not met, the system must be re-tuned and DFTPP re-injected until relative abundances meet criteria.

5.6.10 A chromatogram, spectrum, and mass listing are generated for DFTPP that meets criteria. The recorded injection time of DFTPP that meets criteria is the beginning of the 12-hour time period (or 12 hour clock) for the analysis of standards and/or samples.

5.6.11 In addition, an assessment must be made of GC column performance and injection port inertness by examining the peaks for Pentachlorophenol, Benzidine, and 4,4'-DDT in the tuning check solution analysis. No
excessive tailing should be seen on the Pentachlorophenol or the Benzidine peaks. Excessive tailing is defined as the intensity of the base peak ion at 10 seconds after the apex is no more than 5% of the intensity of the apex. Degradation of DDT to DDD and DDE shall not exceed 20%. Corrective action in the form of injector port cleaning or pre-column maintenance may be required if excessive tailing and/or degradation is seen.

5.6.12 Initial and Continuing Calibration Standards - The following describes steps taken in performing an initial and/or a continuing calibration. As per the client, the target analytes may be all or a subgroup of the compounds listed in the method. If no target analyte list is specified by the client, the laboratory will analyze samples for the compounds listed in Table 4.

5.6.12.1 The following operating conditions are used for the analysis of all standards and samples.

Mass Spectrometer: Electron Impact Ionization Mode
70 V nominal electron energy
One second/scan from 40 - 550 amu
Transfer line: 280º C
Source Temp: 230º C

Gas Chromatograph: Injectable Temp: 250º C
Start Temp: 40º C
Hold Time: 2 minutes
Ramp Rate 1: 20º C/minute to 130º C, hold 0 minutes
Ramp Rate 2: 15º C/minute to 250º C, hold 0 minutes
Ramp Rate 3: 10º C/minute to 310º C, hold 5 minutes

GC temperature program may be different with above parameters depending on columns, project requirements, target compounds list, and so on.
5.6.12.2 For routine analysis, each GC/MS system must be calibrated at a minimum of five concentrations to determine instrument sensitivity and the linearity of the GC/MS response for the semivolatile target compounds.

(a) The concentrations routinely analyzed are 80ng/µL, 60ng/µL, 40ng/µL, 25ng/µL, 10ng/µL, and 5ng/µL per 1µL injection.

5.6.12.3 After completion of acquisition of standards, open Environmental Data Analysis Software. Be sure to have the Bnalist.M or the corresponding processing method loaded. This "method" has been created by lab personnel and contains information regarding tune criteria, compound spectra, retention times, and response references. The ions selected for quantitation (unless there is interference) are those listed in Table 3 of Method 8270D.

(a) Select Quant, Calculate and Generate Report.

(b) Make necessary integration changes by selecting the Qedit option under the Quant option in the menu bar.

(c) Update calibration levels from the Update levels option under Initial on the menu bar.

(d) Select Report Response Factors to Screen under the Initial option on the menu bar. A table with calculated response factors for each compound at each level will appear on the screen. The response factor is calculated as per 9.3.4.1 of the Method 8270D [or as shown in section 5.9.1 of this document] using the peak area for the characteristic ion for each compound.

5.6.12.4 The average RF should be calculated for each compound [section 5.9]. The %RSD [section 5.9] should be less than or equal to 20% for each of the compounds.
compounds must meet minimum response factor listed in Table 4 of the Method 8270D. If criteria are met and time remains in the 12-hour clock, samples may be analyzed.

(a) If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and failed to meet the minimum correlation coefficient (0.990) for alternate curve fits, clean the GC/MSD system and repeat the initial calibration procedure.

(b) If the %RSD of any target analyte exceeds 20% over the calibration range, a regression equation that does not pass through the origin may be employed as long as the coefficient of the determination (COD) is greater than or equal to 0.990.

5.6.12.5 A check of the calibration curve must be performed once every 12-hour time period. This is accomplished by first analyzing a 50ng injection of DFTPP. The ion abundances must meet the criteria as stated in section 5.7.5.

(a) Analysis of the mid-level [CC] standard (e.g. 25ng/µL for targets and surrogates; 20ng/µL for internal standards) is required. The percent difference [%D] shall be calculated for each compound. The %D should be less than or equal to 20% criteria for each target compound for the initial calibration to be valid. If acceptance criteria are met, sample analysis may begin.

Note: Projects that require NELAC protocol to be followed, the initial calibration curve shall be verified using a mid-point standard (ICV) prepared from a second source each time a fresh ICAL curve is prepared. When a second source is not available or there are time constraints that prohibit the purchase of a second source in a timely manner, a person other than the one preparing the calibration standards shall prepare an ICV.
solution from the same source materials. The value determined from the ICV should be within 30% of the expected concentration. Additionally, at least once a year a different CC standard level will be used.

(b) Internal standard area response shall not differ by more than +100% or -50%, as compared with the mid-point standard from the most recent initial calibration.

5.6.12.6 If at any point, criteria are not met, the GC/MS system must be evaluated and corrective action taken as required. This may involve installing a new analytical column or thoroughly cleaning the injector (if the problem seems to be poor performance), or cleaning the ion source collector, ion volume, or lenses. It is the responsibility of the analyst to evaluate and determine the cause of non-compliant performance and make corrections as required before proceeding to sample analysis.

5.7 Procedure

5.7.1 Sample Analysis

5.7.1.1 Locate and remove sample extracts from appropriate freezer and allow to come to room temperature. Transfer a 200µL aliquot of sample extract into an injection vial. Add 4µL of internal standard (1000ng/µL) to each of the samples including any and all QC samples.

5.7.1.2 Set up analysis sequence.

5.7.1.3 An extraction blank will be analyzed after the calibration verification standard in order to ensure that the GC/MSD system is free of contamination. If the method blank indicates contamination, a solvent blank spiked with internal standard shall be analyzed in subsequent sequences to show no carryover and/or contamination from the instrumentation.
5.7.1.4 After acquisition of sample(s) is completed, open the Environmental Data Analysis Software. Select “Quantitate using initial Cal RFs” under the Quant option on the menu bar.

5.7.1.5 To generate quan reports for each sample, select “Calculate and Generate” report under the Quant option of the menu bar. Multiple files may be processed by selecting Tools, Do-list, Summary Quant w/o calc. no print. (Screen will request selection of files for processing.)

5.7.1.6 As with the standards files, manual integrations may be required for missed or incorrectly integrated compounds in the quantitation report of the sample(s). Select Quant, Edit on the menu bar to affect changes needed to the quan report.

5.7.1.7 Positive identification of target analytes will be made using criteria as given in section 7.6 of the Method 8270. A hardcopy of the sample and standard spectrum will be included in the data package.

5.7.1.8 Up to ten peaks not associated with the calibration standard (i.e. - surrogates, internal standards, etc) will be tentatively identified and quantitated.

5.7.1.9 Internal standard area responses will be validated. Area responses shall not differ by more than +100% or -50% as compared with the most recent calibration verification standard. If outside criteria, the instrument shall be checked for malfunctions. Additionally, spiking calculations will be verified and if no errors are found, the poor response may be due to matrix interference and should be reanalyzed. If the majority of the samples in an SDG fail to meet area and/or retention time criteria due to suspected matrix interference, the laboratory shall reanalyze only one or two samples to show proof of matrix interference assumption.
5.7.1.10 If any quantitated analyte exceeds the calibration range, the sample extract shall be diluted appropriately and reanalyzed.

5.7.1.11 A Selected Ion Monitoring (SIM) technique may be used if quantitation limits are lower than the normal range of EI Mass Spectrometry. The 12-hour time period begins at the moment of injection of the first initial calibration standard or a continuing calibration standard. The GC/MS system must be calibrated at a minimum of five concentrations, prior to the analysis of samples and required blanks, to determine instrument sensitivity and linearity. The RF of each target compound must be greater than or equal to the compound’s minimum acceptable RF list in Table 4 of Method 8270D. The %RSD of each target compound should be less than or equal to 20% or meet the minimum correlation coefficient (0.990) for alternate curve fits.

5.8 Calculations

5.8.1 The response factors [RFs] for each target analyte relative to the corresponding internal standards are calculated as follows:

\[ RF = \frac{A_s \times C_{is}}{A_{is} \times C_s} \]

Where,

- \( A_s \) – Peak area of the analyte or surrogate.
- \( A_{is} \) – Peak area of the corresponding internal standard.
- \( C_s \) – Concentration of the analyte or surrogate.
- \( C_{is} \) – Concentration of the corresponding internal standard.

5.8.2 The mean response factor, standard deviation, the %RSD, and %D are calculated as follows:

5.8.2.1 Mean response factor (mRF)
## Test/Analytical Procedure

**01.0403 High Resolution Mass Spectrometry**

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<th>Procedure Number</th>
<th>Revised By:</th>
<th>Title:</th>
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<td>TAP-01-0403-003</td>
<td>Qingchu Peng</td>
<td><em>Method 8270 Analysis of Semivolatile Organic Compounds by combined Gas Chromatography/Mass Spectrometry (GC/MS)</em></td>
</tr>
</tbody>
</table>

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### 5.8.2.2 Standard Deviation (SD)

\[
SD = \sqrt{\frac{\sum_{i=1}^{n} (RF_i - mRF)^2}{n - 1}}
\]

### 5.8.2.3 Relative Standard Deviation expressed as a % (%RSD)

\[
% \text{RSD} = \frac{SD}{mRF} \times 100
\]

### 5.8.2.4 Percent Deviation (%D)

\[
% \text{D} = \frac{RF_i - RF_c}{mRF} \times 100
\]

### 5.8.3 The concentration of the target analytes in the samples are calculated as follows:

\[
C_s = \frac{A_s \times C_{is}}{A_{is} \times mRF}
\]

### 5.8.4 ChemStation files cannot be re-processed once a data package is submitted unless the original file has been backed up already.

#### 5.9 Method Performance

**5.9.1 Method performance shall be monitored through the use of surrogate and matrix spike compounds. Recoveries will be used to generate statistical data for control charting as indicated in section 5.6.7.**
5.10 Pollution Prevention

Pollution Prevention will be performed in accordance with section 4.1-I of the SwRI Safety Policies and Procedures Manual (SPPM).

5.11 Waste management is performed in accordance with the requirements of TAP-01-0407-005, Disposal of Hazardous Material, and TAP-01-0407-008, Disposal of Non-Hazardous Material.

6.0 MAINTENANCE

6.1 GC Maintenance

6.1.1 Daily GC maintenance is performed to ensure generation of reliable and reproducible results.

6.1.2 Oven, injector, interface, and transfer line temperatures should be cooled down before maintenance on the GC is performed. If the GC is also equipped with liquid nitrogen, close valve to tank.

6.1.3 Once the GC is cooled, the following steps may be taken:

6.1.3.1 Replace the quartz liner in injector port.

6.1.3.2 Change the septum in injector nut.

6.1.3.3 Swab the inside of the injector port with methylene chloride.

Note: Caution must be taken in performing this step as methylene chloride vapors, if inhaled, may cause dizziness.

6.1.3.4 Cut and discard the pre-column (~1 meter) and replace with an equal length cut from an unused similar analytical column.
6.1.4 Once maintenance is finished, reset temperatures to original values. Reopen valve to liquid nitrogen tank, if applicable.

6.1.5 More extensive maintenance is performed on an as needed basis. This may include a cleaning of the source and/or the quadrapole rods. Additionally, repair to hardware components may also be required. All such instances of repair/maintenance shall be recorded in an instrument maintenance logbook.

7.0 SAFETY

7.1 In all cases, the Material Safety Data Sheet must be made available to the laboratory personnel. It is the responsibility of the individual user to consult the Material Safety Data Sheet and take any precautions necessary in the usage of a chemical. As a bare minimum, all standards must be prepared in a hood, and safety glasses and lab-coats must be worn at all times when working with neat chemicals. In many cases, it is also recommended that disposable gloves be worn, and all glassware used in the preparation of the standard be disposed of immediately in a proper fashion. In no case is any piece of glassware that has come in contact with a neat chemical or stock solution to be left unlabeled on an open counter. No unlabeled containers of any kind are to be stored in the chemical storage freezers.

7.2 Safety shall be performed in accordance with requirements of CHP-008, Chemical Hygiene Plan for Chemistry and Chemical Engineering Division.

8.0 RECORDS

8.1 Instrument Logbook

8.1.1 BNA GC/MS analysis records are documented on a daily basis in the appropriate instrument logbook. This logbook also will contain instrument operating conditions and all maintenance and/or repairs, as well as difficulties or special circumstances noted with each analysis. Each logbook entry will be signed and dated by the responsible analyst. In addition, all logbook pages associated with a specific case will be
reviewed and signed by another analyst before the data is turned over to the sample management group for data entry.

8.1.2 All documentation in instrument logbooks shall be in black non-soluble ink. Corrections shall be made by crossing a single line through the error and entering the correct information.

8.1.3 Corrections will be initialed and dated. No information shall be obliterated or rendered unreadable.

8.2 Data Reports

Data reports for standards and samples are generated using automated search routines. The resulting data reports are checked by the analyst and corrections are made as required. Corrections on the data report are initialed and dated by the analyst.

8.3 Records

8.3.1 All analytical procedures are recorded in proper laboratory notebooks in accordance with client/contract requirements.

8.3.2 Records generated by the processes of this procedure will be maintained in accordance with Division 01 SOP-01-4.2.4, Storage and Maintenance of Quality Records.

Correction
06-08-11 Corrected the number sequence from section 5.7 to 5.11

Revision 7
Entire document revised to incorporate changes from version D of the EPA method.

Revision 6
Updated section 5 to reflect current procedure practices.

Revision 5
Revised references, 5.4.1, 5.6.6, 5.7, 5.10
Revision 4
TAP revised to expand the summary of the test method
Step 5.6.1 revised to include new first paragraph for further detail concerning Laboratory method blank
Removed references to inactivated procedures.
The solution was brought up to a final volume of 250 mL with a mixture of Acetone/MeOH/DCM (60/30/10).
<table>
<thead>
<tr>
<th>DFTPP MASS ION ABUNDANCE CRITERIA</th>
<th></th>
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<tr>
<td>51</td>
<td>10 to 80% of Base Peak</td>
</tr>
<tr>
<td>68</td>
<td>&lt; 2% of mass 69</td>
</tr>
<tr>
<td>70</td>
<td>&lt; 2% of mass 69</td>
</tr>
<tr>
<td>127</td>
<td>10 to 80% of Base Peak</td>
</tr>
<tr>
<td>197</td>
<td>&lt; 2% of mass 198</td>
</tr>
<tr>
<td>198</td>
<td>Base Peak, or &gt; 50% of Mass 442</td>
</tr>
<tr>
<td>199</td>
<td>5 to 9% of mass 198</td>
</tr>
<tr>
<td>275</td>
<td>10 to 60% of Base Peak</td>
</tr>
<tr>
<td>365</td>
<td>&gt; 1.0% of mass 198</td>
</tr>
<tr>
<td>441</td>
<td>present but &lt; 24% of mass 442</td>
</tr>
<tr>
<td>442</td>
<td>Base Peak, or &gt; 50% of mass 198</td>
</tr>
<tr>
<td>443</td>
<td>15 to 24% of mass 442</td>
</tr>
</tbody>
</table>

The criteria in this table comes from EPA Method 8270D. Alternate tuning criteria may be employed (e.g., CLP or Method 625), provided that method performance is not adversely affected.
This solution was brought to a final volume of 8 ml.

<table>
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<tr>
<th>STD ID</th>
<th>Desired Final conc ng/µL</th>
<th>Final Vol µL</th>
<th>AMT of 80 ng/µL used</th>
<th>AMT of IS (1000 ng/µL) added</th>
<th>DCM Added To Volume</th>
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<td>500</td>
<td>10</td>
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<tr>
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<tr>
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<td>1000</td>
<td>312</td>
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<tr>
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<td>10</td>
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<td>437.5</td>
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### Base/Neutral Fraction

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<th>Acid Fraction</th>
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<tr>
<td>1,4-Dichlorobenzene</td>
<td>2,4-Dichlorophenol</td>
</tr>
<tr>
<td>Hexachlorobutadiene</td>
<td>2-Nitrophenol</td>
</tr>
<tr>
<td>N-Nitroso-di-n-phenylamine</td>
<td>Phenol</td>
</tr>
<tr>
<td>Di-n-octylphthalate</td>
<td>Pentachlorophenol</td>
</tr>
<tr>
<td>Fluoranthene</td>
<td>2,4,6-Trichlorophenol</td>
</tr>
<tr>
<td>Benzo(a)Pyrene</td>
<td></td>
</tr>
</tbody>
</table>

### SPCC Compounds

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

### Table 2 - Surrogate Recovery Limits

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<th>Surrogate</th>
<th>Low/Medium Water</th>
<th>Low/Medium Soil</th>
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<tr>
<td>Nitrobenzene-d5</td>
<td>35-114</td>
<td>23-120</td>
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<tr>
<td>2-Fluorobiphenyl</td>
<td>43-116</td>
<td>30-115</td>
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<td>p-Terphenyl-d14</td>
<td>33-141</td>
<td>18-137</td>
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<td>Phenol-d6</td>
<td>10-94</td>
<td>24-113</td>
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<tr>
<td>2-Fluorophenol</td>
<td>21-100</td>
<td>25-121</td>
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<tr>
<td>2,4,6-Tribromophenol</td>
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### Table 3 - Matrix Spike Recovery Limits

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<th>RPD Soil</th>
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<td>2-Chlorophenol</td>
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<tr>
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<tr>
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<td>51-28-5</td>
<td>2,4-Dinitrophenol</td>
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<td>100-02-7</td>
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<tr>
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<td>1,4-Dichlorobenzene</td>
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<td>2,4-Dinitrotoluene</td>
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<tr>
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Figure 1

File: C:\ELINK\FINN8\DATA\100302\810032T2.D
Operator: SA
Acquired: 3 Oct 2002 14:33 using AcqMethod METHOD.M
Instrument: FINN8
Sample Name: SWRI FINN8 DFTPP
Misc Info: DFTPP TUNING SOLUTION #236-10-01-02
Vial Number: 1

Southwest Research Institute Proprietary
Figure 1A

DPTPP Tune Evaluation

Data File: C:\ELINK\FINN\DATA\100302\810032T2.D  
Acq On:  3 Oct 2002 14:33  
Sample: SWRI FINN DPTPP  
Misc: DPTPP TUNING SOLUTION #236-10-01-02  
MS Integration Params: RTEINTEGRATOR  
Method: C:\ELINK\FINN\QUANT\8270.M (RTE Integrator)  
Title:  8270C/CLP BNA CALIBRATION CURVE

Peak Apex is scan: 670 (7.65 min)
Average of 3 scans: 669,670,671 minus background scan 650 (7.52 min)
## Figure 1B

Average of 7.641 to 7.653 min.: 810032T2.D

**Modified: subtracted**

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Average of 7.641 to 7.653 min.: 810032T2.D

**Modified: subtracted**

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<td>7751</td>
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</table>

Average of 7.641 to 7.653 min.: 810032T2.D

**Modified: subtracted**

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<th>abund.</th>
<th>m/z</th>
<th>abund.</th>
<th>m/z</th>
<th>abund.</th>
<th>m/z</th>
<th>abund.</th>
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<td>2612</td>
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</tbody>
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