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**LABORATORY SOP FOR EPA METHODS 625 AND 8270C:
SEMI-VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS
SPECTROMETRY (GC/MS): CAPILLARY COLUMN TECHNIQUE**

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1. Scope and Application

Methods 625 and 8270C are used to determine the concentration of semi-volatile organic compounds in extracts prepared from liquids, sediments, and a variety of multi-phase samples. The operating procedures described here are designed to meet or exceed the requirements for methods 625 and 8270C. The laboratory currently analyzes the following compounds:

1,4-Dioxane	Isophorone	2-Nitroaniline
Pyridine	2-Nitrophenol	Dimethylphthalate
n-Nitrosodimethylamine	2,4-Dimethylphenol	Acenaphthylene
2-Picoline	Bis(2-chloroethoxy)methane	2,6-Dinitrotoluene
Methylmethanesulfonate	Benzoic acid	3-Nitroaniline
Ethylmethanesulfonate	2,4-Dichlorophenol	Acenaphthene
Aniline	1,2,4-Trichlorobenzene	2,4-Dinitrophenol
Benzaldehyde	aa-Dimethylphenethylamine	4-Nitrophenol
Phenol	Naphthalene	Dibenzofuran
Bis(2-chloroethyl)ether	4-Chloroaniline	Pentachlorobenzene
2-Chlorophenol	2,6-Dichlorophenol	2,4-Dinitrotoluene
1,3-Dichlorobenzene	Hexachlorobutadiene	1-Naphthylamine
1,4-Dichlorobenzene	Caprolactam	2-Naphthylamine
Benzyl alcohol	N-Nitroso-di-n-butylamine	2,3,4,6-Tetrachlorophenol
1,2-Dichlorobenzene	4-Chloro-3-methylphenol	Diethylphthalate
2-Methylphenol	2-Methylnaphthalene	Fluorene
Bis (2-chloroisopropyl)ether	1,2,4,5-Tetrachlorobenzene	4-Chlorophenyl phenyl ether
Acetophenone	Hexachlorocyclopentadiene	4-Nitroaniline
4-Methylphenol	2,4,6-Trichlorophenol	Diphenylamine
N-Nitroso-di-n-propylamine	2,4,5-Trichlorophenol	4,6-Dinitro-2-methylphenol
Hexachloroethane	1,1'-Biphenyl	N-Nitrosodiphenylamine
Nitrobenzene	2-Chloronaphthalene	
N-Nitrosopiperidine	1-Chloronaphthalene	

Phenacetin
Hexachlorobenzene
Atrazine
4-Aminobiphenyl
Pentachlorophenol
Pronamide
Pentachloronitrobenzene
Phenanthrene
Anthracene
Carbazole
Di-n-butyl phthalate
Fluoranthene
Benzidine
Pyrene
p-Dimethylaminoazobenzene
Butylbenzylphthalate
Benzo[a]anthracene
3,3'-Dichlorobenzidine
Chrysene
Bis(2-ethylhexyl)phthalate
Di-n-octylphthalate
Benzo[b]fluoranthene
Benzo[k]fluoranthene
7,12- Dimethylbenz(a)anthracene
Benzo[a]pyrene
3-Methylchloranthrene
Dibenz(a,j)acridine
Indeno[1,2,3-cd]pyrene
Dibenz[a,h]anthracene
Benzo[g,h,i]perylene
Alpha-BHC
Gamma-BHC
Beta-BHC
Delta-BHC
Heptachlor
Aldrin
Heptachlor epoxide
Endosulfan 1
Dieldrin
p,p'-DDE
Endrin
Endosulfan 2
p,p'-DDD
Endrin aldehyde
Endosulfan sulfate
p,p'-DDT
1,2-Diphenylhydrazine
4-Bromophenyl phenyl ether

Surrogate Standards:

2-Fluorophenol
Phenol-d5
Nitrobenzene-d5
2-Fluorobiphenyl
2,4,6-Tribromophenol
Terphenyl-d14

Uncontrolled Copy

- 1.1 The following EPA SW-846 extraction methods are used to prepare samples for 8270C analysis:

3520C, 3541, 3550B & 3580A

- 1.2 Each of these extraction methods uses similar principles. A sample of water or solid is mixed with methylene chloride, which is collected and concentrated to a much smaller volume under controlled conditions. The resulting extracts are then analyzed for the compounds listed in section 1.0
- 1.3 This method describes chromatography conditions that will allow for the GC separation of the compounds in the extract and for their qualitative and quantitative analysis by Mass Spectrometry.

2. Definitions

- 2.1 Refer to Chapter 3 of the Georgia EPD Laboratory Quality Assurance Manual for Quality Control Definitions.

3. Interferences

- 3.1 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Determine if the source of interference is in the preparation and/or cleanup of the samples. Take corrective action to eliminate the problem. Contamination by carryover can occur whenever high concentration and low concentration samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed out between samples with solvent (Methylene Chloride). Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of pure solvent to clean any cross contamination in the system.
- 3.2 Interferences by phthalate esters can pose a major problem in semi-volatile analysis. Common flexible plastics are a primary source of phthalate contamination. Care must be taken during the extraction process to minimize this source of contamination.

4. Safety

- 4.1 Refer to Laboratory Safety, Chemical Hygiene Plan, and Fire Safety Plan.

5. Apparatus and Equipment

- 5.1 Gas Chromatographs
 - 5.1.1 An Agilent gas chromatograph oven 6890 or 7890 is used containing a 30 meter HP 5MS column with a diameter of 0.32 mm and a film thickness of 0.5 μ m or equivalent.
 - 5.1.2 A 30 meter HP 5MS Column with a diameter of 0.25 mm and a film thickness of 0.50 μ m or 1.0 μ m can be used also.
- 5.2 An Agilent autosampler is used to do auto-injection of the sample into a split/splitless injector with a glass sleeve containing no glass wool. The effluent of the column is plumbed directly into the ion source of the mass spectrometer.
- 5.3 The mass spectrometer is an Agilent 5973 or 5977 mass selective detector using an ion pressure gauge, and turbo pumps connected to an Edwards rough pump.
- 5.4 A computer with HP EnviroQuant Software, network capability, and sufficient speed to analyze raw data and present it in a chromatographic report.
- 5.5 Syringes in several volumes ranging from 5 μ L to 1000 μ L are needed for sample dilutions and internal spiking of all extracts.
- 5.6 Sample amber sample vials with graduations in 0.5 mL increments, screw caps, teflon sealed septa for sample removal without opening the vial to the atmosphere. Extra caps

- 5.7 should be available to reseal sample extracts after the septa has been punctured for analysis.
5 and 10 mL volumetric flasks with ground glass stoppers.

6. Reagents

6.1 Standards

- 6.1.1 All standards that are received must have a receiving date stamped on the original box. In addition, all standards must have an expiration date on either the box or ampule. A certification data sheet must be shipped with the standards and is filed in the QC standards logbook. Currently, the internal standards, calibration curve standards, and DFTPP are ordered from several suppliers. Absolute is the primary source with different lot numbers used as secondary sources.
- 6.1.2 Internal standards are stored at room temperature in a dark, dry, cool place in the lab. The glass vials are sealed, and are not opened until ready for use. Internal standards that are opened for use are recorded into the standard logbook with all batch and date made information. A number is assigned to the standard beginning with the GC/MS laboratory number (7) and then the next number available in the standard logbook. After opening the vial, the contents are transferred into a new clean amber autosampler vial, identified with the lab lot number, and sealed with the new screw top lid. The minimum amount of time the vial is open to the atmosphere the better, as the solvent evaporates very quickly.
- 6.1.3 The sealed calibration curve standards are stored at the temperature required by the manufacturer until ready for use. Let the standards come to room temperature before opening the glass sealed vials for preparation of the curve solutions. Any remaining standard left in the vial is stored in new, clean, amber autosampler vial and placed in the freezer. The curve solutions are stored in new, clean, amber autosampler vials and placed in the freezer, after spiking with internal standard. Be sure all vials have proper identification for traceability and expiration dates. Be sure all standards (seven calibration and all daily CCV standards) have individual numbers recorded in the standards logbook to provide proper identification and traceability.
- 6.1.4 The DFTPP stock is stored at ambient temperature. After opening the glass sealed vials for preparation of the DFTPP the remaining solution is stored in new, clean, amber autosampler vials, properly identified, and placed in the freezer.
- 6.1.4.1 The DFTPP solution is purchased in prepared vials with an expiration date. No vial is to be used after its expiration date. The DFTPP solution in the vial is prepared and diluted to a concentration of 50 ppm in methylene chloride. The remainder of the stock solution is stored in a Teflon capped screw top vial in the freezer. The 50 ug/ml solution is also stored in a Teflon capped screw top vial in the freezer.

DFTPP PREPARATION

1. Add 4000uL of MeCl₂ in a class A 5mL volumetric flask.
 2. Add 500 uL of the purchased 500ug/ml DFTPP stock solution, add MeCl₂ to volume.
 3. The resulting DFTPP solution will be 50ug/ml, or, 50ng/uL (1uL is injected)
- 6.1.5 The expiration dates of all standards should be checked to ensure the expiration date has not been exceeded. Any standards past the expiration date should be discarded. All standards that have been opened can be kept in the freezer a maximum of one year.
- ### 6.2 Solvents

- 6.2.1 Methylene Chloride, residue/reagent grade or better.
- 6.2.2 Acetone, reagent grade

7. Sample Collection

- 7.1 Water samples for semivolatile organic compounds are collected in a 1-liter narrow mouth glass bottle. Sample bottles must be cooled to 4°C after sample collection. Two to four bottles are required for each sample. Samples must be extracted within 7 days and the extracts stored at $\leq 10^{\circ}\text{C}$ protected from light, then analyzed within 40 days.
- 7.2 Soil and sediment samples for semi-volatile organic compounds are collected in 8 oz wide mouth glass sample bottles. Sample bottles must be cooled to 4°C after sample collection. Two bottles (4 for QC) are required for each sample. Samples must be extracted within 14 days and the extracts stored at $\leq 10^{\circ}\text{C}$ protected from light, then analyzed within 40 days.

8. Calibration

- 8.1 Calibration Standards Preparation: Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.
 - 8.1.1 Initial Calibration standards are prepared from a number of stock mixes purchased from one of several suppliers. The following will outline the use of Absolute mixes because, at present this is our primary supplier for these standards.
 - 8.1.2 All of these BNA and Benzoic Acid ampules are at a concentration of 2000 ug/ml. Surrogate mixes are also added and have a concentration of 4000 ug/ml.
 - 8.1.3 Preparation of the initial curve must take place under a hood. Proper safety is paramount when handling these standard mixes. A lab coat, gloves, and safety glasses are necessary due to the concentrations and number of hazardous compounds involved.
 - 8.1.4 Take a 10 milliliter volumetric flask and tape it down to the counter in a hood. It is extremely easy to tip these small flasks over.
 - 8.1.5 Be sure all steps are recorded in the standard preparation section of the 8270 log book. Open each ampoule and transfer the contents of each mix into a screw cap extraction vial with a transfer pipette. Label each vial appropriately. Take 1000 uL of each standard mix and 500 uL of the surrogate mix and transfer them into the 10 mL volumetric flask as quickly and carefully as possible. Use dedicated syringes for this measurement. Fill the flask to volume with methylene chloride, again using a dedicated syringe, and cap immediately. Mix thoroughly. Transfer to 4 or 5 new auto sampler vials and store in the freezer until ready to make the 7 BNA dilutions for the curve. The final concentration for the BNA stock standard is 200 ug/ml. The 7 Benzoic Acid curve standards are prepared by directly transferring the purchased stock standard at 2000ug/ml to 1 mL volumetric flasks.
 - 8.1.6 Internal Standards comes at the correct concentration in the ampule. Therefore, simply open an ampule and transfer the contents into a screw cap extraction vial. Store at the manufacturer required temperature in a dark place.
 - 8.1.7 From the stock BNA solution prepare the following concentrations placing volumes given into a cleaned 2 ml volumetric flask. The Benzoic Acid is prepared by itself in a cleaned 1ml volumetric flask.

Final concentration of 7-point standard	Volume of Stock Standard	Volume of Internal Standard	Volume of Methylene Chloride
10 ug/ml (BNA)	100 uL	20 uL	1880 uL
20 ug/ml(BNA)	200 uL	20 uL	1780 uL
50 ug/ml(BNA)	500 uL	20 uL	1480 uL
80 ug/ml(BNA)	800 uL	20 uL	1180 uL
100 ug/ml(BNA)	1000 uL	20 uL	980 uL
120 ug/ml(BNA)	1200 uL	20 uL	780 uL
160 ug/ml(BNA)	1600 uL	20 uL	380 uL
20ug/ml(Benzoic Acid)	10uL	10 uL	980 uL
50ug/ml(Benzoic Acid)	25uL	10 uL	965 uL
100ug/ml(Benzoic Acid)	50uL	10 uL	940 uL
150 ug/ml(Benzoic Acid)	75uL	10 uL	915 uL
200 ug/ml(Benzoic Acid)	100uL	10 uL	890 uL
250 ug/ml(Benzoic Acid)	125uL	10 uL	865 uL
300 ug/ml(Benzoic Acid)	150uL	10 uL	840 uL

- 8.1.8 Mark each vial with the proper concentration and store the vials in the freezer until ready to run a curve.
- 8.1.9 Prepare several 50 ug/ml standards to use as a continuing calibration standard (daily CCV). This standard will be used every day and be subjected to constant heating, cooling, and exposure to light. It will degrade over time, so watch for degradation of problem compounds such as benzidine, pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, 4-chloro-3-methylphenol and benzoic acid. Note, however, these compounds also will degrade if the system is dirty. If it is suspected that the standard is degrading, analyze the 50 ug/ml initial calibration standard and compare it to the daily CCV. If both show poor response for these or other compounds, the system is dirty and the standard is ok. If the initial cal standard is ok, the continuing calibration standard is degrading and should be discarded. A new one should be prepared.
- 8.1.10 A second source standard (ICV) must also be prepared to verify the concentrations of the primary standard used to make the initial curve. This standard can be from another supplier or the same supplier with a different lot number. It is analyzed after each new curve. It should have all the compounds of interest (CCC and SPCC) and be prepared at 50 ug/ml. The percent deviation must be $\pm 30\%$ for all performance compounds (CCC and SPCC).
- 8.1.11 A second initial calibration curve must be prepared for organo-chlorine pesticides and SOW compounds. Absolute is the primary source for pesticides and for SOW compounds. Prepare a 200 ug/ml stock standard by adding 1000 uL of each standard mix and transferring them into the 10 mL volumetric flask.
- 8.1.12 The curve preparation is similar to the preparation of the curve in 8.1.7, in a 2 ml volumetric flask:

Final concentration of 7-point Standard	Volume of Stock Standard	Volume of Internal Standard	Volume of Methylene Chloride
10 ug/ml	100 uL	20 uL	1880 uL
20 ug/ml	200 uL	20 uL	1780 uL
50 ug/ml	500 uL	20 uL	1480 uL
80 ug/ml	800 uL	20 uL	1180 uL
100 ug/ml	1000 uL	20 uL	980 uL
120 ug/ml	1200 uL	20 uL	780 uL
160 ug/ml	1600 uL	20 uL	380 uL

8.1.13 A second source standard must also be prepared for pesticides/SOW to verify concentrations of the primary standards. Prepare at 50 ug/ml.

8.1.14 Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.

8.2 Tuning Standard

8.2.1 A standard containing a mixture of DFTPP (decafluorotriphenylphosphine), 4',4'-DDT, pentachlorophenol, and benzidine is prepared at a concentration of 50 ug/ml. (See 6.1.4.1)

8.3 Calibrating the Instrument

8.3.1 If the Mass Spec is not tuned, then before any analysis, including a calibration, the Mass Spec must be "tuned". A tune consists of the software running a special program, which optimizes the settings of the Mass Spec.

8.3.2 After tuning, a DFTPP standard mix must be analyzed at the beginning of each 12 hour run to verify that the system is still operating correctly. The sequence set up for an analysis run should always have this analysis first. It runs under the DFTPP method and is a short analysis because it is focused on the analysis of a short list of compounds.

8.3.3 The following table gives the correct values that EPA requires for a Mass Spec to be considered "tuned" before each 12 hour sequence:

<u>Mass</u>	<u>Ion Abundance Criteria</u>	<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30-60% of mass 198	199	5-9% of mass 198
68	< 2% of mass 69	275	10-30% of mass 198
70	< 2% of mass 69	365	> 1% of mass 198
127	40-60% of mass 198	441	Present but less than mass 443
197	< 1% of mass 198	442	> 40% of mass 198
198	Base peak, 100% relative abundance	443	17-23% of mass 442

8.3.4 When the tune check has been analyzed, Enviroquant will print out a chromatogram and table similar to the above that will determine if the tune "passed". If all the criteria have "pass" the tune is still valid. If any ion abundance fails, analyze another tune check standard. If it also fails, some type of system cleanup is probably in order and should be performed before any further analyses are made.

8.3.5 Check DDT for % recovery. It is included in the tune mix and should be checked for "breakdown" into its components of DDD and DDE. If the loss of DDT is 20% (of 50 ug/ml) or more, the system is not clean and should have maintenance performed. Usually

this involves changing the glass injection insert and/or gold seal. The calculation for breakdown is a feature of QEdit under ChromEval.

- 8.3.6 Also check the “tailing” of pentachlorophenol and benzidine. This is also a feature of QEdit under ChromEval. The values for acceptance are Benzidine < 3.0 and Pentachlorophenol < 5.0.
- 8.3.7 When the tune criteria check (DFTPP) passes, analyze each of the 7 concentrations of curve standards for the 8270 target compounds. When that has been completed, enter the results into the initial calibration section of EnviroQuant Software. See Section 10.2 for entering data.
- 8.3.8 There are several criteria that must be satisfied before a curve can be considered “valid”. Several compounds fall into two groups that must pass specifications. These are the System Performance Check Compounds (SPCCs, marked with a P in EnviroQuant Software) and Calibration Check Compounds (CCCs, marked with a C in EnviroQuant Software). The list below gives the requirements for these groups:

<u>SPCC compound</u>	<u>Minimum RF</u>
N-nitroso-di-n-propylamine	0.05
Hexachlorocyclopentadiene	0.05
2,4-Dinitrophenol	0.05
4-Nitrophenol	0.05
<u>CCC compound</u>	<u>Maximum RSD</u>
Acenaphthene	30%
1,4-Dichlorobenzene	30%
Hexachlorobutadiene	30%
Diphenylamine	30%
Di-n-octyl phthalate	30%
Fluoranthene	30%
Benzo(a)pyrene	30%
4-Chloro-3-methylphenol	30%
2,4-Dichlorophenol	30%
2-Nitrophenol	30%
Phenol	30%
Pentachlorophenol	30%
2,4,6-Trichlorophenol	30%

- 8.3.9 All compounds of interest should have a %RSD of 15% or less to be considered linear for quantitation purposes. If any target (except the CCC compounds which must always have %RSD of <15%) compound has a higher %RSD it can be checked for Correlation Coefficient (CORR also called R²) using linear or quadratic regression. If the CORR is 0.990 or greater, the curve for that compound is still considered linear and it’s curve can be used for quantitation. This is a feature of Enviroquant under InitCal/Edit Compounds. Further, if the average %RSD of all the compounds of interest is < 15%, the curve is considered valid. (SW-846 Method 8000B Section 7.5.1.2). However, the analyst must take into consideration that the error for calculating compounds with % RSDs higher than 15% is greater, care should be taken when reporting critical data results for any hits associated with high RSDs. See section 11, Calculations.
- 8.3.9.1 The curve should be analyzed by using the evaluation feature of Enviroquant. Go to Initial Cal in the top bar and look for Response Factors to Printer. Choose this and the software will automatically calculate the average response factor and the %RSD for all compounds.

A copy of this must be reviewed an associate or manager and then filed.

- 8.3.10 Relative retention time (RRT) for each compound in each calibration standard should agree within ± 0.06 RRT units. See section 11, Calculations.
- 8.3.11 The calibration for each compound of interest must be verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for the samples. This is accomplished by analyzing a calibration standard (CCV) at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis should meet the verification acceptance criteria listed below:
1. Each SPCC in the standard must meet a minimum response factor of 0.050.
 2. Each CCC %D must be $< 20\%$. See Section 11, Calculations
 3. Internal standards' retention times cannot change by more than 30 seconds from the mid-point standard retention times of the initial calibration curve.
 4. Internal Standard Response must be within -50% to $+100\%$ from the mid-point standard of the initial calibration curve.
 5. Compounds should have a %D of $\pm 20\%$. It is permissible to have 5% of BNA and Pesticide compounds fail $> -20\%$ and there are no positive hits for those compounds. If more than 5% fail the $> -20\%$ D criteria, run another CCV and examine the compounds according to the verification acceptance criteria. Rerun CCV one time, if correct use sample results. If not, correct problem then repeat initial calibration verification and reanalyze all samples since last successful calibration verification. If the CCV fails again, perform maintenance, rerun another CCV, if fails, recalibrate.
- 8.3.12 The Extraction Blank for a batch of extracted samples is usually the first sample analyzed. However, on occasion, samples will be diluted and analyzed without the blank.
- 8.3.13 If all of the above "passes", the system has a valid calibration and analysis of samples can begin. Note that 12 hours after the tune criteria check has been injected, another passing tune check, CCV, and blank must be injected before samples can be analyzed. If any sample extracts are injected after the 12 hour cut off, the sample must be re-analyzed within a valid clock time.

9. Quality Control

- 9.1 The QC parameters to be followed for analysis of semi-volatiles are listed below for initial calibration and daily calibrations. The most important points that must be met before sample analysis can occur are that for every 12 hour analysis period a 50ng sample of DFTPP must pass, and that the 50 ug/ml Daily Standard must meet criteria for the System Performance Check Compounds (SPCC) and the Continuing Calibration Compounds (CCC).
- 9.2 For the daily standard, retention times for target peaks cannot be more than 30 seconds off from the previous day and the internal standard areas cannot vary more than -50% to $+100\%$. The SPCC's must have a minimum response factor of 0.050. In addition, the CCC's must not have a deviation of more than 20% from the initial calibration curve. The performance and continuing calibration criteria listed in the method must be met before analysis can begin.
- 9.3 Surrogates
- 9.3.1 The percent recovery of these compounds is laboratory determined but a starting point is listed in the 625 and 8270C methods.
- 9.3.2 All chromatograms with failed surrogates are examined to determine the reason for the failure and what action is required for reporting the sample. After each batch, calculations of the percent recovery of surrogate standards and the response of the internal standard are

calculated using QABatch in the EnviroQuant software. Using control charts surrogate recovery limits are updated annually, and charted semi-annually.

9.4 Calibration Curve

9.4.1 The initial 7-point calibration curve must meet the criteria listed in the method for Percent Relative Standard Deviation, the System Performance Check Compounds (SPCC), and the Continuing Calibration Compounds (CCC). This criteria must be met or the instrument is considered too reactive for the curve to be valid for analysis. First, the SPCC's must have a minimum average response factor of 0.050. Second, the CCC's must have an average Relative Standard Deviation of less than 30%. If these two measures are not met, then the GC and/or the MS must be examined for problems and the curve re-run. The maximum life of a calibration curve should not exceed 3 months.

9.5 Sample Analysis Batch

9.5.1 Method 625 and 8270 also requires additional QC samples that are extracted and analyzed. These are a method blank, laboratory control sample, laboratory control sample duplicate matrix spike sample, and a matrix spike sample duplicate.

9.5.2 These QC samples are extracted with the samples that need 625 and 8270 analysis, up to a maximum of 20 samples per batch. The group of samples and the QC samples are all together given the name "batch".

9.6 Method Blank

9.6.1 The method blank is extracted with a batch and is used as a quality control measure for the extraction process. This is used to check the analysis results and prove no laboratory contamination of the samples has occurred. Its contents are exactly what the name implies, it is a DI water blank with surrogates and internal standards added. Any 8270 compounds that are detected in a blank must be taken into consideration before the results are reported for the sample.

9.7 Laboratory Control Sample, LCS Duplicate, Matrix Spikes and MS Duplicates

9.7.1 The LCS and LCSD consist of an aliquot of a clean matrix similar to the sample matrix and have the same weight or volume. The LCS and LCSD compounds include the base/neutrals (Acenaphthene, 1,4-Dichlorobenzene, 2,4-Dinitrotoluene, N-nitroso-di-n-propylamine, Pyrene, and 1,2,4-Trichlorobenzene) and the acids (2-Chlorophenol, 4-Chloro-3-methylphenol, 4-Nitrophenol, Pentachlorophenol, and Phenol), all spiked at 100 ppm. Control limits are updated semi-annually.

9.7.2 Matrix Spikes and Matrix Spike Duplicates involve adding a known amount of known compounds that are target compounds of 625 and 8270 analysis to a sample. A batch having matrix spikes will contain a sample extraction, and two other sample aliquots. Each of these two additional samples, are spiked with the same compounds and at the same levels as the LCS and LCSD. The two spiked sample results are tabulated and compared against each other and against the known amounts that were added to the sample originally. The percent recovery for the spike and accuracy are calculated and compared to the LCS control limits. Any failure requires a thorough evaluation of the data by comparison with the LCS and may require that a standard be made up using that analyte and re-extracted and re-analyzed. Control limits are updated semi-annually.

9.8 Sample Data Analysis Records Keeping

9.8.1 Printouts of the DFTPP pass criteria, the daily standard quantitation report, and the continuing calibration report of the daily standard are required for future reference. The 7-point curve percent relative standard deviation report is saved along with the 7 quantitation reports of the 7 concentration levels. A hard copy of each sample analysis report is also stored away. This will include the quantitation report, the triple plot report, and the library search report. The data that is stored on a designated hard drive of the computer system and

is backed-up onto a DVD for storage, and allows for the raw data file to be accessed at a future date. Each batch of samples that is extracted and analyzed is assigned a unique QC batch number by the LIMS system. Here, the results of the blank, the sample duplicate results and the matrix and matrix duplicate results for the samples are stored. The LIMS system also contains the results of the calculations between the LCS and LCSD analysis, and the calculations of the MS and MSD. The QC required LCS, matrix and surrogate recovery calculations are also stored.

9.9 Method Performance

9.9.1 **Method Detection Limit (MDL)** - This is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte. The actual MDL varies depending on the operator accuracy and precision, instrumentation, and matrix. Note that MDL spikes are extracted exactly like samples before analysis.

9.9.1.1 For operational purposes, the MDL should be determined by multiplying the appropriate one-sided 99% T-statistic by the standard deviation obtained from a minimum of seven replicates of matrix spike containing the analytes of interest at a concentration one to five times the estimated MDL. The T-test values are obtained from the table (along with MDL calculation) found in 40CFR-Chapter I-Part 136-Appendix B. This information was used to create the current MDL study for semi-volatiles and is performed once a year. The results can be found in the cabinet for the method MDL in the GC/MS area. There are 7 samples run using a T-test value of 3.14. The calculations include Mean Concentration, Mean Accuracy, Standard Deviation, Relative Standard Deviation, Calculated MDL, and Minimum Instrument Reported MDL.

9.9.1.2 After MDLs are established for the instrument the sample results are reported five times above the MDL level. This is called the Estimated Quantitation Limit (EQL) or Reporting Limit (RL) and is defined as the lowest concentration that can be reliably achieved within specified limits during routine laboratory operating conditions. Precision and accuracy of MDLs are calculated and reported with MDL results on the spreadsheet.

9.10 **Precision and Accuracy** - When starting a new analysis, an Individual Demonstration (IDL) must be run. This involves taking four to seven vials that contain the concentrations of the analyte above the MDL usually at mid-range. Precision is determined by means of replicate analyses. The calculation of precision is determined from the Relative Percent Difference (RPD) of the concentrations (not recoveries) for replicate analysis. The recoveries of most compounds should fall within the established control range and this range should be used as a guide in evaluating in-house performance. Information on Precision and Accuracy can be found in the Quality Assurance Plan. See section 11 of the SOP for calculation formulas.

9.11 **Surrogate Studies** -Surrogate recovery upper and lower limits are made for each of the soil, water, and waste analysis. This involves taking a minimum of 20 random sample surrogate compound results and calculating (P+3S) and (P-3S). P = the average of each surrogate. S = the standard deviation of the percent recovery. The new ranges should fall within the ranges given in the Code of Federal Regulations, Title 40, Part 136, Revision 2, July 1996. The latest, at this revision's date, are listed in Table 13.2.

9.11.1 In addition, every 6 months charting for each surrogate standard per sample matrix is generated using data from all samples. This chart plots the amount of the surrogate recovered and the recovery upper and lower limits. The SQC charts must meet the general criteria laid out in the 625 and 8270C methods.

9.12 To establish the ability to do 625 and 8270C analysis, initial and continuing demonstrations of proficiency (Accuracy, Precision, and Blind Studies) by the analyst must be

demonstrated once every six months with the instrument. This shows proficiency by generating data of acceptable accuracy.

10. Procedure

10.1 Determination of DFTPP -Before any samples or any analysis can occur on the GC/MS, the MS instrument must be proven to be tuned according to EPA 625 and 8270 methodologies. The mass spectrometer must pass the criteria listed below for a 50 ng direct injection of DFTPP. This will demonstrate that the instrument is tuned and will be in effect for a 12 hour period after which the instrument must pass DFTPP again for analysis to continue.

THIS MUST PASS BEFORE ANYTHING CAN RUN ON THE INSTRUMENT !!!!

<u>Mass</u>	<u>Ion Abundance Criteria</u>	<u>Mass</u>	<u>Ion Abundance Criteria</u>
51	30-60% of mass 198	199	5-9% of mass 198
68	< 2% of mass 69	275	10-30% of mass 198
70	< 2% of mass 69	365	> 1% of mass 198
127	40-60% of mass 198	441	Present but less than mass 443
197	< 1% of mass 198	442	> 40% of mass 198
198	Base peak, 100% relative abundance	443	17-23% of mass

10.1.1. Running the DFTPP method - To run the DFTPP method, a 1 µl size sample is injected into the GC column inlet. At the concentration of DFTPP prepared as described in 6.1.4.1, this is the 50 ng sample required by the method to demonstrate that the mass spectrometer is tuned correctly. This tune is valid for only 12 hours, so any samples run must be done in the 12 hour window, or a new DFTPP tune must be run and passed for the next 12 hour period.

Here's how to run this sequence:

1. Remove the DFTPP vial from cold storage and allow it to equilibrate to room temperature prior to analysis.
2. Place the DFTPP vial into tray position number 1..
3. Open the sequence menu again and select "edit sample log table". In the window, change the file name to match today's date and select "OK".
4. Open the sequence menu again and select "position and run"
Select OK for the next window, and then in the next window change the path to match today's date.
5. The resulting DFTPP tune criteria: pass/fail, will be printed out by the program.

10.1.2 Failure of the DFTPP run

If the DFTPP evaluation run fails, the first possible solution is to check the area count of the peak. If it is low, the injector port may be clogged, the column clogged, the needle clogged, or maybe it was just a bad injection requiring a re-injection. If nothing can be found in the area peak or shape to be abnormal, try manually integrating the peak and doing a DFTPP tune evaluation on the mass spectrum. If this also fails, and will not pass, then adjust make small adjustments to the voltage for the repeller, or ion focus lens, or entrance lens. If this fails a re-tuning of the mass spec may be required. The tune file is saved, and the DFTPP re-run. If all this is done and the tune still fails, it indicates that the ion source in the mass spec may be dirty. The mass spec must be disassembled and the ion source cleaned. This cleaning will take about one entire day, and the instrument must pump out overnight to remove all water and air in the mass spec. With this new tune the

daily standard will fail so a new curve will have to be rebuilt. If it passes, sample analysis can take place.

- 10.2 Running the 7 concentrations to build a calibration curve for 625 and 8270C compounds.
There are 3 types of calibration method curves that have to be updated based on sample type: Water, Soil, and Waste. The 7 concentrations are used to update all 3 types of methods, but the units are different for each. Water is ug/L, Soil is ug/Kg, and Waste is mg/Kg.
- 10.2.1 Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.
- 10.2.2 Run the 14 BNA concentrations as 14 samples and quantitate, (the 7 BNA vials and the 7 Benzoic Acid curve concentrations).
- 10.2.3 Make sure each compound has the correct retention time and area. (Run Qedit). In Qedit it is helpful to click on "Spectrum" and "Display Spectrum References." This puts the reference spectrum of the target compound under the scanned spectrum. If the spectrum match it is properly identified.
- 10.2.4 Load the midpoint to update retention times and areas of target compounds in the software.
- 10.2.5 Open the "InitCal" window and select "Update Levels".
- 10.2.6 Select "Recalibrate".
- 10.2.7 Select the calibration level ID for the sample loaded.(For a 50ug/ml sample, select ID 50).
- 10.2.8 Select "replace" for responses and retention times.
- 10.2.9 Select "do update".
- 10.2.10 For the Benzoic Acid curve concentrations the area for each curve level and internal standard are loaded into the Chemstation software.
- 10.2.11 Load the next highest sample concentration, update that level and repeat for the remaining levels to build the curve. Open file menu and select "Save Method".
- 10.2.12 Open the "InitCal" window and select "Response factors to printer".
At this point, important quality control checks are reviewed and **MUST** pass before the curve can be used for quantitation of unknown samples.
- 10.2.13 The first quality control check is that the System Performance Check Compounds (SPCC) that must have a minimum average response factor of 0.050, if this fails on any one, then the instrument is deemed too reactive for analysis, and the GC and/or MS must be examined and the problem corrected, and the curve re-run. The SPCC's are: N-Nitroso-di-n-propylamine; Hexachlorocyclopentadiene; 2,4-Dinitrophenol; and 4-Nitrophenol. These compounds must have an average Relative Standard Deviation of no more than 15%.
- 10.2.14 The second quality control is the Calibration Check Compounds (CCC). The CCC must have an average Relative Standard Deviation of no more than 30%. If the CCC does, then the GC and/or the MS must be examined for problems, and the curve re-run. The CCC's are: Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, N-Nitrosodiphenylamine, Di-n-octylphthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol, and 2,4,6-Trichlorophenol.
- 10.3 Running the Pesticide and SOW 7-point Curve
- 10.3.1 Remove sample bottles, standards, and reagents from cold storage and allow them to equilibrate to room temperature prior to sample preparation and/or analysis.
- 10.3.2 Run the 7 concentrations as 7 samples and quantitate.
- 10.3.3 Make sure each sample has correct times and response areas (no duplications or omissions of times or areas).
- 10.3.4 Load the midpoint (50 ug/ml) sample.
- 10.3.5 Open the InitCal window and select "Update Levels" in the window.

- 10.3.6 Select "Recalibrate".
- 10.3.7 Select the calibration level ID for the sample loaded. (Ex. For a 50ug/ml sample, select ID 50P)
- 10.3.8 Select "replace" for responses and retention times.
- 10.3.9 Select "Do Update".
- 10.3.10 Load the other curve concentrations, and repeat for the remaining levels to build the curve.
- 10.3.11 Open the file menu, and select "Save method".
- 10.3.12 The 7 point curve calibration for the pesticides is now complete.

10.4 Daily Operations

- 10.4.1 Run the DFTPP solution - The DFTPP method will cause a 1 µl sized sample to be injected into the GC, this is the 50 ng sample required by the method to prove the mass spec instrument is tuned. This tune is valid for only 12 hours, so any samples run must be done in the 12 hour window, or a new DFTPP tune must be run and passed for the next 12 hour period. Here's how to do a run sequence:
 1. Remove the DFTPP vial from cold storage and allow it to equilibrate to room temperature prior to analysis.
 2. Place the DFTPP vial into the tray.
 3. Open the sequence menu and select "edit sample log table". In the window, change the file name to match today's date and select "OK". (Example 041500t1 for April 15th, 2000).
 4. Open the sequence menu again and select "position and run".
 5. Select OK for the next window, and then in the next window change the path to match today's date. (Example C:\HPCHEM\DATA\041500).
 6. The resulting DFTPP tune criteria pass/fail will be printed out by the program.
- 10.4.2 Run the daily standard - This section assumes that the DFTPP has passed and that the 7-point curve has already been built and is valid with the RF's all within the criteria. This 50 ppm daily standard is a middle range concentration sample that contains the BNA target compounds that are analyzed for 625 and 8270 semivolatiles by EPD laboratories. An additional 22 compounds are not included in this daily standard analysis.
 1. Remove the DFTPP vial from cold storage and allow it to equilibrate to room temperature prior to analysis.
 2. Place the 50 ppm daily standard vial into tray position number one.
 3. Open the sequence menu and select "Edit Sequence".
 4. From the sequence window. Open the sequence menu again and select "edit sample log table".
 5. In the window, change the file name to match today's date and select "OK"
Example: C:\HPCHEM\DATA\980709s1. Open the sequence menu again and select "position and run".
 6. Select **OK**, and in the next window change the **file** path to match today's date.
 7. The results form will be printed out by the program.
 8. If there are any problems, try "EasyID" or "Qedit" to fix it and requantitate.
 9. Analyze the 50 ppm standard with the additional 22 compounds in the same manner as above.
- 10.4.3 Apply the daily standard to the continuing calibration - The daily standard contains certain compounds that **MUST PASS** 625 and 8270C criteria before analysis can occur of any sample. This criteria of passing is valid only for the next 12 hours marked after the DFTPP tune was injected and passed. The first set of compounds that must pass with the daily standard are the system performance check compounds (SPCC). The SPCC must have a minimum response factor of 0.050. These compounds are: N-Nitroso-di-n-propylamine,

hexachlorocyclopentadiene, 2,4-dinitro-phenol, and 4-nitrophenol. The second set of compounds that must pass are the calibration check compounds (CCC). The CCC must not have a deviation of more than 20% from the initial calibration curve. These compounds are: Acenaphthene, 1,4-Dichlorobenzene, Hexachlorobutadiene, N-Nitrosodiphenylamine, Di-n-octylphthalate, Fluoranthene, Benzo(a)pyrene, 4-Chloro-3-methylphenol, 2,4-Dichlorophenol, 2-Nitrophenol, Phenol, Pentachlorophenol, 2,4,6-Trichlorophenol. All compounds should have a plus or minus 20% or less deviation. See 10.4.4. A copy of the daily CC report must be filed with all other QC data. In addition, another copy of the daily CC report must be given to the manager with the samples that ran in the 12 hour period to verify good quality control work for proper validation.

1. From the top window, open "data analysis"..
2. Open the "**file**" menu, choose "load data **file**", then select the file for today's daily standard run.
3. Open the "concal" menu and select "evaluate **file** as continuing cal to printer"
4. If the printout has no out of range RFs, then the instrument is good and ready to collect data.
5. The continuing calibration is now complete.

10.4.4 Failure of the daily standard evaluation - The daily standard (50 ug/ml), has several criteria for "passing" the current calibration curve. The % deviation is 20 percent for the CCCs, a 0.05 minimum response for the SPCCs, and internal standard responses between -50% and +100% of the average responses of the initial curve. If the standard fails two attempts, there may be many causes, and it is the experience of the instrument operator that will determine how much time and effort is involved in determining and correcting the problem. The simplest solution is to run QEdit and try to integrate the peaks that have failed, then re-evaluate the data file. If the sample fails again, routine maintenance is the next step. This usually includes replacing the glass injector insert and cutting a small amount of column on the injector side, but this may not help in solving the problem. The most effective solution is to cut 6 inches from the injector side of the column, as this area receives the brunt of all samples injected. Another solution is to replace the gold disk which forms the bottom of the injector cavity seal. This disk is replaced only when necessary, and only after all other options have been exhausted. Other solutions may involve replacing the injector septum, and in a final effort after all avenues have been exhausted, then a new curve must be built, with the RFs falling within the acceptance criteria of the method. If this fails, and is repeated, and no other problem can be found, then a new column must be installed and the curve built. One other solution is to clean the mass spec ion source and try to rebuild the curve. Note, that if only certain compounds are failing, in particular, benzidine, pentachlorophenol, or other phenols (acid compounds), it is not usually a dirty source. This is an indication of active sites (dirt) somewhere in the injector (insert or gold seal) or in the beginning 6 inches to 1 foot of column. A dirty source will usually give low responses for all compounds and/or noise at the baseline during an analysis.

10.4.5 Set up and run samples - The samples are removed from the freezer and allowed to come to room temperature.. Ten microliters (10 uL of the internal standard are added to each sample, be it the blank, duplicate, matrix spike, etc. If a sample has been concentrated to the volume greater than 1 mL, then a 1 mL aliquot is taken in a clean vial, and the 10 uL of internal standard is added to this vial. Measure internal standard spikes carefully. The internal standard response will GREATLY affect results.

1. Open the sequence menu and select "Load".
2. From the sequence window of choices select the last date a sequence has been run. These will be in the format of mmddyy or yymmdd.

3. Open the sequence menu, again, and select "Edit sample log table".
4. Enter the vial position on the autosampler, filename, and information, then select "file" and "save as". Save it to the current date.
5. Open the sequence menu, again, and select "Position and run".
6. Select "OK", and in the next window change the file path to match today's date .
7. Select "OK" and then "file" and "save". Save as the same name given above and when asked if it is desired to overwrite the file select "yes".
8. Open the sequence menu, one more time and select "Position and Run" again. Choose the first position to be analyzed, and select "run sequence" in the next window.
9. When asked if key words should be recognized, select "no".
10. The sequence will start now.

The sample will be injected onto the instrument, and when finished, the chromatogram and report data files will be printed. Note that the data has "QC not reviewed" at the top of the chromatogram reports. This means that the report has not been "Q-edited".

10.5 Data Integration

- 10.5.1 Q-edit. The qualitative identification of compounds determined by the 625/8270 method is based upon retention time and on comparison of the mass spectra of the sample. Also used are a background subtraction correction and characteristic ions in a reference mass spectrum. The characteristic ions from the reference mass spectrum are the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. For samples containing compounds not associated with the calibration standard a library search is used for the purpose of tentative identification. Once a compound has been identified it will undergo a quantitative analysis based on the integrated abundance of the characteristic ion vs. the nearest internal standard abundance.
- 10.5.2 All chromatographic data must be Q-edited. Load the data analysis part of Enviroquant software from the Icon on the desktop. Go into file and load the data file of interest.
- 10.5.4 When the chromatogram is loaded, go into Quant and choose "QEdit". The program will load a blow up of the ion chromatograph for the primary ions of each compound. A small box will appear over the chromatogram with a list of all the compounds in the target list.
- 10.5.4 Left double-click on the first compound which is an internal standard. The peak chosen to be this compound will be in the chromatogram window and there should be a small "x" by the name in the smaller box just picked the compound from. Look for a red line near the bottom of the peak in the chromatogram window. It should be reasonably straight going from "valley to valley" of the peak. If it is at a severe angle or only covers half the peak, move the mouse cursor to the beginning of the peak, hold down the right mouse button and drag the cursor under the peak to where it ends. Release the mouse button and it should draw a straight line under the peak.
- 10.5.5 Look for the next compound that has an "x" by it or if it is an internal standard (an "i" is next to the name when highlighted), or surrogate compound. Many times the beginning of the peak will fall slightly outside retention time window and will not be picked up by the software. If a surrogate or internal standard does not have an "x" by it this is usually the case. Double click on the compound with this problem and draw the base line as described above. This will define the peak area and put an "x" by that compound.
- 10.5.6 Check the chromatogram save the changes by going to file and choose save and exit. Note that any change made to a peak's integration will cause a "m" to be placed on the report. Manual integrations will be noted with an "m" on the component line.

10.6 Logbooks

- 10.6.1 A copy of each day's sequence is placed in the green 3-ring binder labeled "instrument run log" for the instrument. Make sure that any analysis run for a day is placed in the book.

- Number each page as it is placed in the run log; initial and date.
- 10.6.2 All maintenance, routine and otherwise, is logged into the red "Maintenance Log Book". In addition, there is a general maintenance section for cleaning and replacing instrument parts.
- 10.6.3 A bound logbook to record the data-file number, name of project, description of sample, volume, and date of run is recommended but not required.
- 10.6.4 All standard lot numbers are issued from an oversized 3 ring black logbook located in the volatiles GC/MS Lab. Take the next available space and write the next sequential number. Note the GC/MS lab is lab #7, so all lot numbers begin with 7-. Fill in the appropriate information and use this lot number to identify the standard.
- 10.6.5 A bound logbook is also recommended for quality control standards such as DFTPP, daily standard, and five-point curve standards, record the data-file number, description of each standard, volume, and date of run. For mixture preparations, record the quality control lot number and preparation instructions. Prepare each mixture according to suppliers instructions.
- 10.7 Currently the GC/MS operating conditions are saved under the Enviroquant software method 8270.m on GCMS SVOL3 and SVOL4. Each time a new initial curve is created, the method should be updated with the name using the date the curve was created. Therefore a record of the method used for earlier data must be kept in case the data is reprocessed several months or years later.
- 10.7.1 GC/MS parameters for GCMS SVOL3 and GCMS SVOL4 are listed below. Note: These may change as conditions warrant, due to column cutting, installation of new column, or other change to the system requiring longer or shorter run times, scan times, etc.:

GCMS SVOL 3 , GCMS SVOL 4	
GC Parameter	Setting
Inlet Temperature	250°C
Source Temperature	230°C
Oven Equib Time	1.5 min
Oven Temp Max	325°C
Initial Temperature	40°C
Hold Time.	2 Min.
Level 1: Rate, Next Temp, Hold Time	35°C/minute, 130°C, 0.00 minutes
Level 2: Rate, Next Temp., Hold Time	12°C/minute, 310°C, 8.0 minutes
Total Run Time	27.57 minutes
Initial Pressure	7.02 psi
Initial Flow	1.0 mL/min
Column Length	30.00 meters
Column Diameter	0.25 millimeters
Gas	Helium

GCMS SVOL 3 , GCMS SVOL 4	
GC Parameter	Setting
Vacuum Compensation	On

Mass Spec Parameters	Settings
Tune File	dftpp.u
Acquisition Mode	Scan
Solvent Delay	varies
EM Relative	True
Low Mass	35
High mass	550
Threshold	150
Sample #	1 A/D Samples 2

10.8 Laboratory Information Management System (Labworks) - After the sample batch has been analyzed it is ready to be reported. The data is uploaded to the Laboratory Information Management System (LIMS). Here are the LIMS test codes for semi-volatiles GC/MS:

\$LA827C* True value of LCS
 \$A_827C* True value of Matrix Spike and Laboratory Control Compounds
 \$827C* Results for samples
 \$B_827C* Results for blanks
 \$D_827C* Results for Matrix Spike Duplicate
 \$LD827C* Results for Laboratory Control Spike Duplicate
 \$LR827C* Recoveries for Laboratory Control Spike
 \$L28270C Recovery for LCSD
 \$LS827C* Results for Laboratory Control Spike
 \$LP827C* Calculation of precision between LCS and LCS Duplicate
 \$P_827C* Calculation of precision between the Matrix Spike and Matrix Spike Duplicate
 \$R_827C* Recoveries of Matrix Spike Compounds
 \$RD827C* Recoveries for Laboratory Control Spike Duplicate
 \$S_827C* Results for Matrix Spike
 \$IA827C* ICV Amount for SVOC
 \$IS827C* ICV Results for SVOC
 \$IR827C* ICV Recovery for SVOC
 *Insert letter for appropriate matrix: W(water), S(soil), or T(waste)

BATCHING ANALYSIS LIST

#Q\$827CW Analysis for 827C Waters
 #Q\$827CS Analysis for 827C Soil
 #Q\$827CT Analysis for 827C Waste

Here are the LIMS test codes for semi-volatile by method 625:

\$LA625 True value of LCS
 \$A_625 True value of Matrix Spike and Laboratory Control Compounds

\$625	Results for samples
\$B_625	Results for blanks
\$D_625	Results for Matrix Spike Duplicate
\$LD625	Results for Laboratory Control Spike Duplicate
\$L2625	Recovery of LCSD
\$LR625	Recoveries for Laboratory Control Spike
\$LS625	Results for Laboratory Control Spike
\$LP625	Calculation of precision between LCS and LCS Duplicate
\$P_625	Calculation of precision between the Matrix Spike and Matrix Spike Duplicate
\$R_625	Recoveries of Matrix Spike Compounds
\$RD625	Recoveries for Laboratory Control Spike Duplicate
\$S_625	Results for Matrix Spike

BATCHING ANALYSIS LIST

#Q\$625 Analysis for 625 Waters

Here are the LIMS test codes for TCLP semi-volatile by method 1311/8270C:

TCLP Samples

TCLPBNA	Semi-volatile TCLP Warranted?
\$TPBNA	Semi-volatile TCLP (1311)
\$A_TPANA	Amount Spiked Semi-volatiles TCLP (1311)
\$LATPBNA	LCS Amount Semi-volatiles TCLP (1311)
\$B_TPANA	Method Blank for 8270 TCLP (1311)
\$LSTPBNA	LCS Results for Semi-volatiles TCLP (1311)
\$LDTPBNA	LCS DUP Results for Semi-volatiles TCLP (1311)
\$S_TPANA	Results for Matrix Spike Semi-volatiles TCLP (1311)
\$D_TPANA	Results for Matrix Spike DUP Semi-volatiles TCLP (1311)
\$LRTPBNA	LCS Recovery Semi-volatiles TCLP (1311)
\$L2TPBNA	LCSD Recovery Semi-volatiles TCLP (1311)
\$LPTPBNA	LCS/LCSD Precision Semi-volatiles TCLP (1311)
\$R_TPANA	Recovery MS Semi-volatiles TCLP (1311)
\$RDTPBNA	Recovery MSD Semi-volatiles TCLP (1311)
%P_TPANA	MS/MSD Precision Semi-volatiles TCLP (1311)

BATCHING ANALYSIS LIST

#Q\$TPBNA

10.8.1 A batch should be created first for the samples of interest. In Method 625 and 8270, batches are actually created in the Sample Extraction Department. An extraction sheet with all the samples listed is provided, beginning and ending volumes or masses, extraction method used, sample used for MS/MSD spiking, etc. These samples will be defined as a "batch" in Labworks. Use the following procedure to create a batch in Labworks:

1. Open "QA Batches" under QA/QC and click on "New Batches"
2. Click on "Batch by sample" and a box will appear. In the box, enter all the sample numbers listed on the extraction sheet. (Highlight the sample numbers and press "ctrl-C" to save the sample numbers which will be needed later in results entry). Then click on "find samples". Observe that the samples numbers are correct.

3. Click on "OK" and the next screen will appear (All sample numbers should say "pending").
4. This screen will list all the analyses that are associated with each sample. Only 625 or \$827C* analyses are checked with a red checkmark. Check or uncheck an analysis by either clicking on the top of the column, which will check or uncheck the entire column or click on each white box to check or uncheck it. Click "OK" when the appropriate boxes are checked.
5. The next screen will give information for batch size. Click "OK".
6. The next screen will give a list of all the samples in the batch under batch name and QC sample ID. The batch number displayed should be written on the batch checklist and/or the extraction blank report sheet for future reference if needed. It is extremely difficult to find what samples are associated with what batches without the batch number. Look for the sample used for MS/MSD on the extraction sheet and left click on that sample number. MS/MSD appear next to the QC sample ID box. This sets it as the QC sample associated with this batch and creates many additional test codes for Labworks that will be fill in later. Click on "OK" and a new batch will be created.
7. Finally the screen will go to a screen with several option buttons on the right side. Choose the "print" option. A box will appear with several report styles listed. Go down the list and look for INSTR. Click on this and a batch sheet will be printed to put with the package. Sign and date the batch sheet.

10.8.2 Samples are uploaded into Labworks by the following procedure:

1. Open Labworks and go into "Results".
2. Enter all the Sample numbers in the box or press "ctrl-V if the sample numbers were saved as instructed above.
3. Click on "OK" and then multi-sample spreadsheet when asked.
4. When the spreadsheet opens it will list each sample number at the top and list all the associated analyses down the side. Look for the appropriate test code, see 10.8.
5. Under each sample number it should say "pending" and have a white background for the sample (\$625 and \$827C*) or QC sample (\$625 and \$827C*).
6. Move the mouse curser on the "pending" box and right click on the box. An insert will open with a group of choices. Pick "modify results" and left click when it is highlighted.
7. Choose "Load" and a screen similar to windows explorer will open. Choose the appropriate computer and hard drive which contains the data. For example go to drive C on computer GCMS SVOL3 or SVOL4. Click on HPchem, then 1, data, then pick the date, listed as a sub directory for the data. A list of all the sample file names will appear, choose the one of interest.
8. Make sure that GC/MS rr file is picked from the choices on the left of the screen. When choosing the sample of interest, double click on the file directory and then the same file name but with a "rr" extension will be listed. Click on this file and then "OK".
9. The previous screen of analytes and MDLs will appear and in a few seconds the results should be loaded. Closely check the results in Labworks with the reports generated during the sample run.
10. Several calculations are necessary now to determine if the results are representative of the sample volume or weight. Labworks has default reporting limits for each analysis. These reporting limits are based on certain volumes or weights of sample. If the sample amount is different than the expected volume or weight the Dilution Factor must be changed to reflect the difference. Waters are based on a 1000 mL sample volume.

Calculation of Dilution Factor:

$$\frac{1000 \text{ mL} \times D}{(\text{amount extracted})} = \text{Dilution Factor}$$

where:

D = dilution ratio (a 2x dilution would be a 2 for the “D”)

The dilution factor is put into Labworks in the upper right hand corner. If the amount extracted is 1000 mLs, and there were no dilutions, the DF is 1. All DF entries are to two significant figures. Note the Dilution Factor only changes the MDL (reporting limits), not any results.

Soils are based on a 30 gram sample weight. Wastes are based on a 2 gram sample weight.

11. Waters - A common problem with water samples is sediment in the liter bottle. This means that less than 1000 mLs is available for extraction.
12. Soils - Soils have a built in problem. Labworks test codes are built around a 30 gram sample amount for extraction per earlier extraction methods. The latest EPA method for soil extractions requires 20 grams. Also, dry weight comes into account. To compensate for varying levels of moisture, all soil samples require a dilution factor to be calculated and used in reporting. Results and reporting limits for soils should be multiplied by the dilution factor before reporting.

Calculation of Dilution Factor:

$$\frac{30\text{g} \times D}{(\text{amount extracted}) \times (\% \text{ solids}/100)} = \text{Dilution Factor}$$

The dilution factor is put into Labworks in the upper right hand corner. If the amount extracted is 30g, and there were no dilutions, the DF is 1. However all soil extractions are usually made with 20 grams wet weight. All DF entries are to two significant figures.

13. Wastes - Fewer corrections are required for waste samples. Samples are reported on wet weight basis so no % solids comes into play. However, many times the sample has such a thick or obviously contaminated matrix and is diluted initially to prevent problems with GPC clean-up and or concentration during sample extraction. This must be taken into account for the sample by the Dilution Factor in Labworks as described above.

Calculation of Dilution Factor:

$$\frac{2\text{g} \times D}{(\text{amount extracted})} = \text{Dilution Factor}$$

The dilution factor is put into Labworks in the upper right hand corner. If the amount extracted is 2g, and there were no dilutions, the DF is 1. All DF entries are to two significant figures.

14. Once raw data has been loaded and the correct dilution factor has been entered, the values that transfer for any positive concentrations may require corrections. It is a guaranteed correction if the samples are soils. Each positive concentration for a target compound or library search compound (see below) will need to be corrected for any dilutions, compensations for change of matrix (soils), or sample extraction problems that required either a final volume greater than 1 milliliter or an initial dilution before GPC

- clean-up. See Section 11 for calculations.
15. Enviroquant software has been set to identify those compounds with peaks that are 100% of the closest Internal Standard. After careful examination of the library search, any spectra that match compounds in the library should be reported by their IUPAC name. The estimated amount of the compound is placed in the concentration box after calculated for any dilutions or soil adjustments. After this amount is entered, a space and the letters TIE are placed next to it for Tentatively Identified Estimated.
 16. If there are any problems with the sample entry QC or Spike compounds, the results will have a red background. This indicates that the compound used as a QC compound, either a spike or a surrogate, fell outside the acceptable range set in Labworks. Note the limit range for the compound. Left click on the amount of the compound. Then right click. A box will appear with all the QC compounds control limits. The compound in question will be highlighted in blue. Jot down these limits next to the surrogate or spike that failed on the quantitation report. Also draw a small arrow next to the results pointed up for "above control limits" and down for "below control limits". Note any deviation from an "in control" sample with a corrective action.
 17. Corrective Actions - Listed below are some of the common problems that occur and require that a corrective action be filled out for the sample:
 1. Surrogate % recovery Failure
 2. Spike Compound % recovery Failure
 3. Precision failure (LCS/LCSD, MS/MSD)
 4. Sample extracted or analyzed past the established EPA recommended holding time from sample collection date.

While there are many others, these are the most common problems. Two steps are required to fill out a corrective action. First, a Corrective Action Form must be filled out. These are located in the "pink" loose leaf binder located in Volatile GC/MS lab. Find the next available blank sheet, usually in the active section. Fill out the form noting the problem or problems. Note that the numbering system consists of 7-mmddy-xx, where 7 is our lab, mm is the month leading with a 0 for January through September (1-9), dd is the day with single digits leading with a 0, and the year is the last two digit of the year. XX is the next available number in the book. Look at the previous number for a corrective action and use the next number. Fill out the corrective action as completely as possible and note the number on the batch checklist sheet in the data package.

Next while the associated sample is open in Labworks with the loaded data, etc. click on "Anl comments". This will open up a text box. Lead off with the test code that is associated with the sample. Then describe the corrective action listing any failing compounds, surrogate or spike, their % recoveries and limits, and an explanation if possible for the failure. Usually this is matrix interference unless it is a blank, LCS or LCSD. Also note if the LCS passed all QC control requirements. Finally end the comment with the corrective action number and "save".

Example:

\$827CW-EPA8270 - Sample had two surrogate compounds, 2-Fluorophenol (11% recovery, limits 25 -94%) and 2,4,6-Tribromophenol, (14% recovery, limits 22 - 131%) with % recoveries outside acceptable control limits due to matrix interferences. LCS QC results were within acceptable control limits.

18. Finally, click on “Save” in the upper right corner. This will enter the data for that sample into a temporary file. When all the samples and QC have been entered for the batch all should have a “done” for the 8270 test codes. Click on the “Save” button in the left top corner. This will permanently enter the results into Labworks. If it is necessary to go back to a “completed” or “done” sample and make a change, enter why there was a change in the text box that appears and save the change. The audit trail notes the analyst making the change and why for any future inquiries.
19. QA/QC Batch Report – This report will contain all QC data for the sample batch. Double click (left) on the QC Batch icon on the desktop. Enter the batch number generated in 10.8.1.8. Left click on Preview the QC report button to see any boldly highlighted data that indicates failed QC. Most highlighted data should have a comment at the end of the QC report. Print two copies of the report for the data package.
20. Site Investigation QC Reports – Site Investigation samples require additional QC data with the data package. This data reports the recovery for the CCV run with the batch that included the site investigation samples.
21. Open “Results” in Labworks to upload \$IA827C for the matrix involved (W, S, or T).
22. Left click on “pending” box, right click and chose “Enter or Load results” by left clicking.
23. Choose “load results”.
24. Go to drive S and select LW300e_c and select MIX.
25. Left click on 8270IA.rr and select OK. This will input the known amount of the CCV (50ppm). Left click on “Store Results”.
26. Upload the results for the CCV as directed above for samples, into \$IS8270 for the matrix involved. \$IR8270 will be calculated by Labworks.
27. Double click on the QC Batch Report icon on the desktop. Click on the “Preview SIHW QC Report” button to review the CCV recoveries. Print two copies of the SIHW QC Report for the data package.
28. This is a brief overview of Labworks data entry. Some results entries are extremely difficult to correct and may require the assistance of an experienced analyst.

11. Calculations

11.1 Daily Calibration Verification and Continuing Calibration

A 50 ppm calibration standard ensures the instrument’s SPCCs and CCCs meet method performance criteria. For any 12-hour analysis period, prior to samples analysis, a one point daily continuing calibration verification is performed. The System Performance Check Compounds (SPCCs) must meet the minimum average relative response factor. For the Calibration Check Compound (CCCs) the percent drift for each CCC is not to exceed 20% of the initial calibration. If the continuing calibration does not meet method performance criteria then the instrument must be recalibrated.

Calculate the percent drift using the following equation:

$$\text{Equation 11.1.1 } \% \text{Drift} = (C_i - C_c) / C_i \times 100$$

where:

C_i = Calibration Check Compound standard concentration.

C_c = Measured concentration using selected quantitation method.

11.2 Relative Response Factor (*RRF*)

Relative Response Factor: Calculate the relative response factors (*RRF*) for each target compound relative to the appropriate internal standard (i.e., standard with the nearest retention time) using the following equation:

$$\text{Equation 11.2.1 } RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

where:

<i>RRF</i>	= Relative response factor
<i>A_x</i>	= Area of the primary ion for the compound to be measured
<i>A_{is}</i>	= Area of the primary ion for the internal standard
<i>C_{is}</i>	= Concentration of internal standard spiking mixture, ppb
<i>C_x</i>	= Concentration of the compound in the calibration standard, ppb

11.3 Mean Relative Response Factor (\overline{RRF})

Mean Relative Response Factor: Calculate the mean RRF (\overline{RRF}) for each compound by averaging the values obtained at the five concentrations using the following equation:

$$\text{Equation 11.3.1 } \overline{RRF} = \frac{\sum_{i=1}^n x_i}{n}$$

where:

\overline{RRF}	= Mean relative response factor
<i>x_i</i>	= RRF of the compound
<i>n</i>	= Number of values

11.4 Percent Relative Standard Deviation (*%RSD*)

Using the RRFs from the initial calibration, calculate the *%RSD* for all target compounds using the following equations:

$$\text{Equation 11.4.1 } \%RSD = \frac{SD_{RRF}}{RRF} \times 100$$

and

$$\text{Equation 11.4.2 } SD_{RRF} = \sqrt{\frac{\sum_{i=1}^n (RRF_i - \overline{RRF})^2}{n - 1}}$$

where:

SD_{RRF}	= Standard deviation of initial response factors (per compound)
RRF_i	= Relative response factor at a concentration level
\overline{RRF}	= Mean of initial relative response factors (per compound).
n	= Number of values

11.5 Relative Retention Times (RRT)

The retention time for each internal standard must be within ± 30 seconds of the retention time of the internal standard in the most recent valid calibration. Relative retention time of each analyte within ± 0.06 RRT units of the RRT. Calculate the RRT s for each target compound over the initial calibration range using the following equation.

$$\text{Equation 11.5.1 } RRT = \frac{RT_c}{RT_{IS}}$$

where:

RT_c	= Retention time of the target compound
RT_{IS}	= Retention time of the internal standard.

11.6 Mean of the Relative Retention Times (\overline{RRT}):

Calculate the mean of the relative retention times (\overline{RRT}) for each analyte target compound over the initial calibration range using the following equation:

$$\text{Equation 11.6.1 } \overline{RRT} = \frac{\sum_{i=1}^n RRT}{n}$$

where:

\overline{RRT}	= Mean relative retention time for the target compound for each initial calibration standard
RRT	= Relative retention time for the target compound at each calibration level
N	= Number of values

Tabulate the area response (Y) of the primary ion and the corresponding concentration for each compound and internal standard.

11.7 Mean Area Response (\bar{Y}) for Internal Standard:

Calculate the mean area response (\bar{Y}) for each internal standard compound over the initial calibration range using the following equation:

$$\text{Equation 11.7.1 } \bar{Y} = \frac{\sum_{i=1}^n Y_i}{n}$$

where:

\bar{Y}	= Mean area response
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Y = Area response for the primary quantitation ion for the internal standard for each initial calibration standard.

11.8 Mean of the Retention Times (\overline{RT}) For Internal Standard:

Calculate the mean of the retention times (\overline{RT}) for each internal standard over the initial calibration range using the following equation:

$$\text{Equation 11.8.1 } \overline{RT} = \sum_{i=1}^n \frac{RT_i}{n}$$

where:

\overline{RT} = Mean retention time
 RT = Retention time for the internal standard for each initial calibration standard.
 n = Number of values

11.9 Percent Difference (%D):

Calculate the percent difference in the RRF of the daily RRF (24-hour) compared to the mean RRF in the most recent initial calibration. Calculate the %D for each target compound using the following equation:

$$\text{Equation 11.9.1 } \%D = \frac{RRF_c - \overline{RRF}_i}{\overline{RRF}_i} \times 100$$

where:

RRF_C = RRF of the compound in the continuing calibration standard
 \overline{RRF}_i = Mean RRF of the compound in the most recent initial calibration.

11.10 Sample Concentration Calculation.

$$\text{Equation 11.10.1 } C_x = \frac{A_x C_{is} DF}{A_{is} RRF}$$

where:

C_x = Compound concentration, ppm
 A_x = Area of the characteristic ion for the compound to be measured
 A_{is} = Area of the characteristic ion for the specific internal standard
 C_{is} = Concentration of the internal standard spiking mixture, ppm
 RRF = Relative response factor from the analysis of the continuing calibration standard or the mid level standard of the initial calibration
 DF = Dilution factor. If no dilution is performed, $DF = 1$

Table 13.1 RLs for EPA 625 and SW-846/8270C

Parameter/ Method	Analyte	Matrix (Aqueous)		Matrix (Soil)	
		RL	Unit	RL	Unit
SVOCs EPA 625/SW-846 Method 8270C	1,4-Dioxane	10	ug/l	660	ug/kg
	Pyridine	10	ug/L	660	ug/kg
	n-Nitrosodimethylamine	10	ug/L	660	ug/kg
	2-Picoline	10	ug/L	660	ug/kg
	Methylmethanesulfonate	10	ug/L	660	ug/kg
	Ethylmethanesulfonate	20	ug/L	660	ug/kg
	Aniline	10	ug/L	1300	ug/kg
	Benzaldehyde	10	ug/L	660	ug/kg
	Phenol	10	ug/L	660	ug/kg
	bis(2-Chloroethyl)ether	10	ug/L	660	ug/kg
	2-Chlorophenol	10	ug/L	660	ug/kg
	1,3-Dichlorobenzene	10	ug/L	660	ug/kg
	1,4-Dichlorobenzene	10	ug/L	660	ug/kg
	Benzyl Alcohol	20	ug/L	1300	ug/kg
	1,2-Dichlorobenzene	10	ug/L	660	ug/kg
	2-Methylphenol	10	ug/L	660	ug/kg
	Bis (2-Chloroisopropyl) ether	10	ug/L	660	ug/kg
	Acetophenone	10	ug/L	660	ug/kg
	4-Methylphenol	10	ug/L	660	ug/kg
	N-Nitroso-di-n-propylamine	10	ug/L	660	ug/kg
	Hexachloroethane	10	ug/L	660	ug/kg
	Nitrobenzene	20	ug/L	1300	ug/kg
	N-Nitrosopiperidine	10	ug/L	660	ug/kg
	Isophorone	10	ug/L	660	ug/kg
	2-Nitrophenol	10	ug/L	660	ug/kg
	2,4-Dimethylphenol	10	ug/L	660	ug/kg
	Bis(2-Chloroethoxy)methane	10	ug/L	660	ug/kg
	Benzoic Acid	50	ug/L	3300	ug/kg
	2,4-Dichlorophenol	10	ug/L	660	ug/kg
	1,2,4-Trichlorobenzene	10	ug/L	660	ug/kg
	aa-Dimethyl-phenthylamine	10	ug/L	660	ug/kg
	Naphthalene	20	ug/L	1300	ug/kg
	4-Chloroaniline	10	ug/L	660	ug/kg
2,6-Dichlorophenol	10	ug/L	660	ug/kg	
Hexachlorobutadiene	10	ug/L	660	ug/kg	
Caprolactam	10	ug/L	660	ug/kg	
N-Nitroso-di-n-butylamine	10	ug/L	660	ug/kg	
4-Chloro-3-Methylphenol	20	ug/L	1300	ug/kg	
2-Methylnaphthalene	10	ug/L	660	ug/kg	
1,2,4,5-Tetrachlorobenzene	10	ug/L	660	ug/kg	
Hexachlorocyclopentadiene	10	ug/L	660	ug/kg	

Table 13.1 RLs for EPA 625 and SW-846/8270C

Parameter/ Method	Analyte	Matrix (Aqueous)		Matrix (Soil)	
		RL	Unit	RL	Unit
SVOCs EPA 625/SW-846 Method 8270C	2,4,6-Trichlorophenol	10	ug/L	660	ug/kg
	2,4,5-Trichlorophenol	10	ug/L	660	ug/kg
	1,1'Biphenyl	10	ug/L	660	ug/kg
	2-Chloronaphthalene	10	ug/L	660	ug/kg
	1-Chloronaphthalene	10	ug/L	660	ug/kg
	2-Nitroaniline	50	ug/L	3300	ug/kg
	Dimethylphthalate	10	ug/L	660	ug/kg
	Acenaphthylene	10	ug/L	660	ug/kg
	2,6-Dinitrotoluene	10	ug/L	660	ug/kg
	3-Nitroaniline	50	ug/L	3300	ug/kg
	Acenaphthene	10	ug/L	660	ug/kg
	2,4-Dinitrophenol	50	ug/L	3300	ug/kg
	4-Nitrophenol	50	ug/L	3300	ug/kg
	Dibenzofuran	10	ug/kg	660	ug/kg
	Pentachlorobenzene	10	ug/kg	660	ug/kg
	2,4-Dinitrotoluene	10	ug/L	660	ug/kg
	1-Naphthylamine	10	ug/L	660	ug/kg
	2-Naphthylamine	10	ug/L	660	ug/kg
	2,3,4,6-Tetrachlorophenol	10	ug/L	660	ug/kg
	Diethylphthalate	10	ug/L	660	ug/kg
	Fluorene	10	ug/L	660	ug/kg
	4-Chlorophenyl-phenylether	10	ug/L	660	ug/kg
	4-Nitroaniline	20	ug/L	1300	ug/kg
	Diphenylamine	10	ug/L	660	ug/kg
	4,6-Dinitro-2-methylphenol	50	ug/L	3300	ug/kg
	N-Nitrosodiphenylamine	10	ug/L	660	ug/kg
	1,2-Diphenylhydrazine	10	ug/L	660	ug/kg
	4-Bromophenyl-phenylether	10	ug/L	660	ug/kg
	Phenacetin	20	ug/L	1300	ug/kg
	Hexachlorobenzene	10	ug/L	660	ug/kg
	Atrazine	10	ug/L	660	ug/kg
	4-Amino-biphenyl	20	ug/L	1300	ug/kg
	Pentachlorophenol	50	ug/L	3300	ug/kg
	Pronamide	10	ug/L	660	ug/kg
	Pentachloronitrobenzene	20	ug/L	1300	ug/kg
	Phenanthrene	10	ug/L	660	ug/kg
	Anthracene	10	ug/L	660	ug/kg
	Carbazole	10	ug/L	660	ug/kg
	Di-n-butylphthalate	10	ug/L	660	ug/kg
	Fluoranthene	10	ug/L	660	ug/kg
Benzidine	10	ug/L	660	ug/kg	

Table 13.1 RLs for EPA 625 and SW-846/8270C

Parameter/ Method	Analyte	Matrix (Aqueous)		Matrix (Soil)	
		RL	Unit	RL	Unit
SVOCs EPA 625/SW-846 Method 8270C	Pyrene	10	ug/L	660	ug/kg
	p-Dimethylaminoazobenzene	10	ug/L	660	ug/kg
	Butylbenzylphthalate	10	ug/L	660	ug/kg
	Benzo[a]anthracene	10	ug/L	660	ug/kg
	3,3'-Dichlorobenzidine	20	ug/L	1300	ug/kg
	Chrysene	10	ug/L	660	ug/kg
	Bis(2-ethylhexyl)phthalate	10	ug/L	660	ug/kg
	Di-n-octylphthalate	10	ug/L	660	ug/kg
	Benzo[b]fluoranthene	10	ug/L	660	ug/kg
	Benzo[k]fluoranthene	10	ug/L	660	ug/kg
	7,12- Dimethylbenz(a)anthracene	10	ug/L	660	ug/kg
	Benzo[a]pyrene	10	ug/L	660	ug/kg
	3-Methylchloranthrene	10	ug/L	660	ug/kg
	Dibenz(a,j)acridine	10	ug/L	660	ug/kg
	Indeno[1,2,3-cd]pyrene	10	ug/L	660	ug/kg
	Dibenz[a,h]anthracene	10	ug/L	660	ug/kg
	Benzo[g,h,i]perylene	10	ug/L	660	ug/kg
	Alpha-BHC	10	ug/L	660	ug/kg
	Gamma-BHC	10	ug/L	660	ug/kg
	Beta-BHC	10	ug/L	660	ug/kg
	Delta-BHC	10	ug/L	660	ug/kg
	Heptachlor	10	ug/L	660	ug/kg
	Aldrin	10	ug/L	660	ug/kg
	Heptachlor epoxide	25	ug/L	1800	ug/kg
	Endosulfan 1	50	ug/L	3300	ug/kg
	Dieldrin	10	ug/L	660	ug/kg
	p,p'-DDE	10	ug/L	660	ug/kg
	Endrin	20	ug/L	1300	ug/kg
	Endosulfan 2	50	ug/L	3300	ug/kg
	P,p'-DDD	10	ug/L	660	ug/kg
Endrin aldehyde	10	ug/L	660	ug/kg	
Endosulfan sulfate	25	ug/L	1800	ug/kg	
p,p'-DDT	10	ug/L	660	ug/kg	

Table 13.2 Acceptance Criteria for Method EPA 625 and SW846/8270

(24 month upper and lower control limits for water and soil, 7/01/12 - 07/01/14)

(36 month upper and lower control limits for waste, 1/01/04 - 12/31/06)

Method 8270C	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)	Accuracy Waste (%R)	Precision Waste (RPD)
SS							
	2-Fluorophenol	10-100	≤ 30%	23-100	≤ 40%	21-100	≤ 50%
	Phenol-d5	10-100	≤ 30%	22-108	≤ 40%	15-109	≤ 50%
	Nitrobenzene-d5	21-102	≤ 30%	22-101	≤ 40%	21-100	≤ 50%
	2-Fluorobiphenyl	30-106	≤ 30%	26-112	≤ 40%	19-108	≤ 50%
	2,4,6-Tribromophenol	10-118	≤ 30%	11-115	≤ 40%	23-109	≤ 50%
	Terphenyl-d14	49-130	≤ 30%	31-130	≤ 40%	44-112	≤ 50%
LCS							
	Phenol	11-100	≤ 30%	28-100	≤ 40%	15-118	≤ 50%
	2-Chlorophenol	23-100	≤ 30%	25-100	≤ 40%	21-110	≤ 50%
	1,4-Dichlorobenzene	23-100	≤ 30%	23-100	≤ 40%	11-108	≤ 50%
	n-Nitroso-di-n-propylamine	31-103	≤ 30%	29-102	≤ 40%	10-131	≤ 50%
	1,2,4-Trichlorobenzene	28-100	≤ 30%	24-100	≤ 40%	13-119	≤ 50%
	4-Chloro-3-methylphenol	40-100	≤ 30%	34-100	≤ 40%	27-111	≤ 50%
	Acenaphthene	43-103	≤ 30%	38-100	≤ 40%	19-119	≤ 50%
	4-Nitrophenol	10-100	≤ 30%	36-100	≤ 40%	17-119	≤ 50%
	2,4-Dinitrotoluene	37-120	≤ 30%	41-101	≤ 40%	18-124	≤ 50%
	Pentachlorophenol	13-110	≤ 30%	32-109	≤ 40%	20-109	≤ 50%
	Pyrene	50-126	≤ 30%	41-114	≤ 40%	22-114	≤ 50%

Note: Upper control limits are set to not less than 100%.

Table 13.3 Summary of Calibration and QC Procedures for Method EPA 625 and 8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 625/ SW846 - 8270C	Semi-Volatile Organics	Five -point initial calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥ 0.050 ; %RSD for CCCs ≤ 30% drift, should be less than 15% for all other analytes. If CORR used, > 0.990.	Correct problem then repeat initial calibration	
		Second-source calibration verification	Once per five-point initial calibration	Analytes within ±30% of expected value for SPCC and CCCs.	Correct problem and then repeat initial calibration.	
		Calibration verification	Daily, before sample analysis, every 12 hours of analysis time	SPCCs average RF ≥ 0.050; and CCCs ≤ 20% drift; and all calibration analytes within ±20% of expected value	Rerun CCC one time, if passes use sample result. If not, correct problem (usually clipping column and changing insert) then repeat calibration verification once. If passes reanalyze all samples since last successful calibration verification. If fails, recalibrate.	

Table 13.3 Summary of Calibration and QC Procedures for Method EPA 625 and 8270C

Method	Applicable Parameter	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria
EPA 625/ SW846 - 8270C	Semi-Volatile Organics	Initial Demonstration: Demonstrate ability to generate acceptable accuracy and precision using four replicate analysis of a QC check sample	Once per analyst	QC limits set for LCS must be used and all tested analytes must fall within these limits for acceptable results.	Recalculate results; locate and fix problem with system and then rerun demonstration for those analytes that did not meet criteria	
		Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to criteria listed in the method description	Retune instrument and verify with DFTPP tune check again.	
		Internal Standards	Immediately after or during data acquisition of calibration check standard	Retention time \pm 30 seconds: EICP area within -50% and +100% of initial calibration midpoint	Inspect mass spectrometry or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Flag all compounds associated with failed internal standard with "J".
		Method Blank	One per analytical batch	No analytes detected >RL	Ensure no contamination then reanalyze method blank and all samples processed with the contaminated blank	If unable to re-analyze, flag with a "B"
		LCS/LCSD for selected analytes	One LCS/LCSD per analytical batch	QC acceptance criteria established by control charts semi-annually. Precision depends on matrix, 30, 40, 50% for waters, soils, wastes	Correct problem then reanalyze the LCS and all samples in the affected batch	If unable to re-analyze, flag with a "J"
		Surrogate spike	Every sample, spiked sample, standard, and method blank	QC acceptance criteria set by control charts semi-annually	Flag Report	If determined that sample matrix is interfering.
		Matrix Spike and Matrix Spike Duplicate	One MS&MSD per analytical batch	QC acceptance criteria set by LCS control charts semi-annually	Flag Report	Matrix interference
		MDL Study	Once per year	Detection limits established shall be <the RLs in SOP		
		Estimated amount for analytes above the 5-pt calibration curve	none	<u>Waters</u> < 160ug/L <300ug/L Benzoic Acid <u>Soils</u> < 8,000ug/Kg <15,000 ug/Kg Benzoic Acid <u>Waste</u> < 8mg/Kg wastes. <15mg/Kg Benzoic Acid	Sample must be diluted and reanalyzed.	Apply E to all analytes out of range.

Appendix A – Quality Assurance Criteria for Method EPA 8270C*

Table A.1 - Quality Assurance Criteria for EPA 8270C Soils

This replaces Table 13.3 in Sop 7-007 Rev 17

Method 8270C	Analyte	Accuracy Aqueous (%R)	Precision Aqueous (RPD)	Accuracy Soil (%R)	Precision Soil (RPD)	Accuracy Waste (%R)**	Precision Waste (RPD)
SS							
	2-Fluorophenol	10-130	≤ 30%	12.8-130	≤ 40%	70-130	≤ 50%
	As Concentration	10-130 µg/L		1280-13000µg/kg			
	Phenol-d5	10-130	≤ 30%	15.2-130	≤ 40%	70-130	≤ 50%
	As Concentration	10-130 µg/L		1520-13000 µg/kg			
	Nitrobenzene-d5	10-130	≤ 30%	13.4-130	≤ 40%	70-130	≤ 50%
	As Concentration	10-130 µg/L		1340-13000µg/kg			
	2-Fluorobiphenyl	10-130	≤ 30%	20-130	≤ 40%	70-130	≤ 50%
	As Concentration	10-130 µg/L		2000-13000µg/kg			
	2,4,6-Tribromophenol	10-130	≤ 30%	10-130	≤ 40%	70-130	≤ 50%
	As Concentration	10-130 µg/L		1000-13000µg/kg			
	Terphenyl-d14	25-130	≤ 30%	28.9-130	≤ 40%	70-130	≤ 50%
	As Concentration	25-130 µg/L		2890-13000µg/kg			
LCS							
	Phenol	12-130	≤ 30%	27-130	≤ 40%	70-130	≤ 50%
	2-Chlorophenol	27-130	≤ 30%	27-130	≤ 40%	70-130	≤ 50%
	1,4-Dichlorobenzene	26-130	≤ 30%	25-130	≤ 40%	70-130	≤ 50%
	n-Nitroso-di-n-propylamine	30-130	≤ 30%	29-130	≤ 40%	70-130	≤ 50%
	1,2,4-Trichlorobenzene	30-130	≤ 30%	31-130	≤ 40%	70-130	≤ 50%
	4-Chloro-3-methylphenol	29-130	≤ 30%	31-130	≤ 40%	70-130	≤ 50%
	Acenaphthene	36-130	≤ 30%	35-130	≤ 40%	70-130	≤ 50%
	4-Nitrophenol	14-130	≤ 30%	36-130	≤ 40%	70-130	≤ 50%
	2,4-Dinitrotoluene	33-130	≤ 30%	39-130	≤ 40%	70-130	≤ 50%
	Pentachlorophenol	37-130	≤ 30%	31-130	≤ 40%	70-130	≤ 50%
	Pyrene	55-130	≤ 30%	45-130	≤ 40%	70-130	≤ 50%
MS							
	Phenol	10-130	≤ 30%	13-130	≤ 40%	70-130	≤ 50%
	2-Chlorophenol	10-130	≤ 30%	16-130	≤ 40%	70-130	≤ 50%
	1,4-Dichlorobenzene	24-130	≤ 30%	10-130	≤ 40%	70-130	≤ 50%
	n-Nitroso-di-n-propylamine	24-130	≤ 30%	13-130	≤ 40%	70-130	≤ 50%
	1,2,4-Trichlorobenzene	26-130	≤ 30%	13-130	≤ 40%	70-130	≤ 50%
	4-Chloro-3-methylphenol	14-130	≤ 30%	21-130	≤ 40%	70-130	≤ 50%
	Acenaphthene	27-130	≤ 30%	24-130	≤ 40%	70-130	≤ 50%
	4-Nitrophenol	10-130	≤ 30%	20-130	≤ 40%	70-130	≤ 50%
	2,4-Dinitrotoluene	28-130	≤ 30%	22-130	≤ 40%	70-130	≤ 50%
	Pentachlorophenol	10-130	≤ 30%	10-135	≤ 40%	70-130	≤ 50%
	Pyrene	40-130	≤ 30%	34-130	≤ 40%	70-130	≤ 50%

*LCS/LCSD MS/MSD recovery and precision limits, and surrogate recovery limits based on control charts of data collected from 1/1/2015 to 12/31/2018.

**Default Limits, after 20 samples, limits will be re-calculated