ESS Laboratory
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SOP NO. 60_8270C
SEMIVOLATILE ORGANIC COMPOUNDS ANALYSIS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY
(EPA Method 625/SW-846 METHOD 8270C)

REVIEWED BY:

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SEMIVOLATILE ORGANIC COMPOUNDS ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (EPA Method 625/SW 846 METHOD 8270C)

1.0 SCOPE AND APPLICATION

1.1 To determine the concentration of semi-volatile organic compounds in extracts prepared from all types of solid waste matrices, soils, and waters. Parameters measured and method reporting limits

	Aqueous	Soil		Aqueous	Soil
Analyte	**ug/L**	**ug/Kg**	**Analyte**	**ug/L**	**ug/Kg**
1,4-Dichlorobenzene-d4	IS	IS	Dimethylphthalate	10	330
N-Nitrosodimethylamine	10	330	Acenaphthylene	10	330
Pyridine	10	330	2,6-Dinitrotoluene	10	330
2-Fluorophenol	Surr	Surr	2-Nitroaniline	10	330
bis(2-Chloroethyl)ether	10	330	Acenaphthene	10	330
Phenol-d5	Surr	Surr	2,4-Dinitrophenol	10	330
2-Chlorophenol	10	330	Dibenzofuran	10	330
Phenol	10	330	4-Nitrophenol	10	330
Aniline	10	330	3-Nitroaniline	10	330
2-Chlorophenol-d4(SURR)	Surr	Surr	2,4-Dinitrotoluene	10	330
1,3-Dichlorobenzene	10	330	Fluorene	10	330
1,4-Dichlorobenzene	10	330	2,3,4,6-Tetrachlorophenol	10	330
1,2 Dichlorobenzene-d4	Surr	Surr	Diethylphthalate	10	330
1,2-Dichlorobenzene	10	330	4-Chloro-phenyl-phenyl ether	10	330
Benzyl Alcohol	10	330	Phenanthrene-d10	IS	IS
bis(2-chloroisopropyl)Ether	10	330	4-Nitroaniline	10	330
2-Methylphenol	10	330	4,6-Dinitro-2-Methylphenol	10	330
Acetophenone	10	330	N-nitrosodiphenylamine	10	330
N-Nitroso-Di-n-Propylamine	10	330	Azobenzene	10	330
Hexachloroethane	10	330	2,4,6-Tribromophenol	Surr	Surr
3+4-Methylphenol	10	330	4-Bromophenyl-phenylether	10	330
Naphthalene-d8	IS	IS	Hexachlorobenzene	10	330
Nitrobenzene-d5	Surr	Surr	Pentachlorophenol	10	330
Nitrobenzene	10	330	Phenanthrene	10	330
Isophorone	10	330	Anthracene	10	330
2-Nitrophenol	10	330	Carbazole	10	330
Benzoic Acid	10	330	Di-n-butylphthalate	10	330
2,4-Dimethylphenol	10	330	Fluoranthene	10	330
bis(2-Chloroethoxy)methane	10	330	Benzidine	10	330
2,4-Dichlorophenol	10	330	Chrysene-d12	IS	IS
1,2,4-Trichlorobenzene	10	330	Pyrene	10	330
Naphthalene	10	330	Terphenyl-d14	Surr	Surr
4-Chloroaniline	10	330	Butylbenzylphthalate	10	330

Hexachlorobutadiene	10	330	3,3'-Dichlorobenzidine	10	330
4-Chloro-3-Methylphenol	10	330	Benzo(a)anthracene	10	330
2-Methylnaphthalene	10	330	Chrysene	10	330
1-Methylnaphthalene	10	330	bis(2-Ethylhexyl)phthalate	10	330
Acenaphthene-d10	IS	IS	Perylene-d12	IS	IS
Hexachlorocyclopentadiene	10	330	Di-n-octylphthalate	10	330
2,4,6-Trichlorophenol	10	330	Benzo(b)fluoranthene	10	330
2,4,5-Trichlorophenol	10	330	Benzo(k)fluoranthene	10	330
2-Fluorobiphenyl	Surr	Surr	Benzo(a)pyrene	10	330
Biphenyl	10	330	Indeno(1,2,3-Cd)Pyrene	10	330
2-Chloronaphthalene	10	330	Dibenzo(a,h)Anthracene	10	330
			Benzo(g,h,i)perylene	10	330
1,2,4,5-Tetrachlorobenzene	10	330	Pentachloronitrobenzene	10	330

IS = Internal standard
Surr = Surrogate

1.2 This method can be used to quantitate most neutral, acidic and basic organic compounds that are soluble in methylene chloride and capable of being eluted without derivatization as sharp peaks from gas chromatographic fused silica column coated with slightly polar silicone.

1.3 Some compounds may require special treatment when being determined by this method. (See Interference and Potential Problems, section 5.0)

2.0 METHOD SUMMARY

2.1 Prior to using this method, samples are prepared for chromatography using the appropriate preparation and cleanup methods. A measured volume or weight of sample (approximately 1 L for liquids, 2 g to 30 g for solids) is extracted using the appropriate sample extraction technique. Liquid samples are extracted at acidic pH with methylene chloride using a continuous liquid-liquid extraction (SOP 50_3520C). Solid samples are extracted with methylene chloride using either manual or automated Soxhlet extraction (SOP 50_3540B or 50_3541), Ultrasonic Extraction (SOP 50_3550B) or Microwave Extraction (SOP 50_3546) methods. A variety of cleanup steps may be applied to the extract, depending on (1) the nature of the co-extracted matrix interferences and (2) the target analytes. After cleanup, the extract is analyzed by injecting a 1-uL sample into a gas chromatograph with a narrow-bore fused silica capillary column and mass spectrometer detector (GC/MS). This method describes the appropriate chromatographic conditions to achieve separation for a qualitative and quantitative GC/MS analysis.

3.0 HEALTH AND SAFETY

3.1 Each employee has been trained and has acknowledged being trained in the safe use and handling of chemicals being used in the laboratory. This training has been performed according to the ESS Training SOP 80_0016 and by the Chemical Hygiene Plan, SOP No. 90_ 001, in conjunction with the Safety orientation.

3.2 All sample and material handling should be done in a hood while using proper protective equipment to minimize exposure to liquid or vapor. Minimum personnel protective equipment includes the use of laboratory safety glasses, a lab coat or apron, and protective gloves.

3.3 The MSDS for the concentrated chemicals used in the area are kept on file in a central location that is available for all employees to review.

3.4 Several chemicals used in this procedure are tentatively classified as known or suspected carcinogens. Extreme caution should be used in handling all chemicals in this procedure.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Aqueous samples are collected in 1 Liter borosilicate glass jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4°C. Two liters should be provided so samples can be re-extracted when necessary. Aqueous samples must be extracted within 7 days from date sampled.

4.2 Soil/sediment samples are collected in 4 – 8 ounce jars with Teflon lined caps. The samples are stored in a dark walk-in cooler at 4°C. Up to thirty grams of sample are required for extraction and ten grams is required to determine the percent solids. One hundred grams should be provided so samples can be re-extracted when necessary. Soil/Sediment samples must be extracted within 14 days of date sampled.

4.3 All extracts are stored in 2 ml Teflon capped vials in the extract storage refrigerator located in the SVOA lab. These extracts are stored at 4°C and must be analyzed within 40 days of date extracted.

5.0 INTERFERENCES AND POTENTIAL PROBLEMS

5.1 Blanks, samples and spikes must be evaluated for interferences. The source of interference should be identified and corrective action should be taken to eliminate the problem.

5.2 Contamination by carry over can occur whenever high concentration and low concentration samples are sequentially analyzed.

5.3 Contamination by phthalate esters can occur during sample preparation. Common flexible plastics contain varying amounts of phthalate esters that are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoid contact with any plastic materials and check all solvents and reagents for phthalate contamination to minimize interferences from phthalate esters. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

5.4 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor. Estimated values are reported at 50ug/L for a 1 liter water extraction and 1650ug/Kg for a 30 gram soil extraction.

5.5 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

5.6 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

5.7 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

5.8 Since 1,2-Diphenylhydrazine oxidizes to Azobenzene in the GC injection port, it is indistinguishable from Azobenzene on the GC/MS and will be reported as Azobenzene.

6.0 EQUIPMENT/APPARATUS

6.1 Gas Chromatography/Mass Spectrometer system

6.1.1 **Gas Chromatography**: HP 5890 series II with EPC (electronic pressure control)

6.1.2 **Column**: J&W DB-5MS, HP-5MS or RESTEK RXI-5MS (30m X 0.25 mm ID with 25 um film thickness) or equivalent.

6.1.3 **Mass Spectrometer**: HP 5971 or HP 5972 capable of scanning from 35-500 amu every 1 second or less, using 70 volts (nominal) electron energy in the EI mode. The MS must be capable of meeting the criteria in Table 2 when 50 ng of DFTPP (Decafluorotriphenylphosphine) is injected.

6.1.4 **GC/MS interface**: Must give an acceptable response at a 50-ng injection and Pass the tuning criteria on Table 2.

6.2 Data System: Data system:

6.2.1 **Computers:** The Semi-Volatiles laboratory has three GC/MS systems analyzing method 625/8270C. SVOA MS1 has an AST computer with a Windows 95 operating system. SVOA MS2 has DELL computers with Windows NT operating system. SVOA MS3 has DELL computers with Windows 95 operating system. All computer systems are networked to a Windows 2000 server, which is the destination of all files. A differential back-up is performed nightly and a full back is performed each weekend using Veritas Backup Exec

to tapes. As the systems acquires and stores data onto the server, the server becomes full. The data is downloaded and archived onto CDs.

6.2.2 **Software:** HP/Agilent Environmental Chemstation - The software is interfaced to the mass spectrometer detectors and allows the continuous acquisition and storage on machine-readable media of all chromatograms obtained throughout the duration of the instrument program. The software is capable of integrating the abundance in any EICP between specified times. Current versions SVOA MS1 and MS3: G1032C version C.01.00 and SVOA MS2: G1701BA Version B.01.00.

6.3 10µL, 25µL, 100µL, 500µL, 1000µL **Hamilton syringes**.

6.4 **Volumetric Flasks**: Class A

6.5 **Balance**: Top loading, OHAUS,® Precision Standard

6.6 **Sample vials**: Glass with Teflon-lined crimp tops

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**: Reagent grade or better chemicals shall be used in all tests. If the purity of a reagent is in question, it is analyzed for contamination. If the target analytes concentration is less than the ½ MRL in the method blank, then the reagent is acceptable.

7.1.1 Organic free reagent water. (ESS DI water system)

7.1.2 Methylene Chloride, HPLC pesticide grade.

7.1.3 Acetone, HPLC pesticide grade.

7.2 **Standards**:

7.2.1 All Primary, Stock, and working standard information is tracked in logbooks according to SOP 60_0001 and also in the Promium Element LIMS.

7.2.2 **Primary Standards**: The certificates of analysis in Attachment A detail the individual analytes and concentrations in each of these standard mixes. The primary standards are purchased as certified mixes and stored in a dark refrigerator at 4 ± 2°C. Primary standards are not used after the manufacturer's expiration date and once opened are not to be used after one year. The refrigerator is located in the SVOA lab. Standards must not be stored with samples.

Standard	Source	Vendor	Catalog No.	Conc. (µg/ml)

8270 Mix #1	Primary	Restek	31850	500-1000
8270 Mix #2	Primary	Restek	31806	1000
8270 Mix #3	Primary	Restek	31825	2000
8270 Mix #4	Primary	Restek	31879	2000
SemiVOA Mix 1	Secondary	Ultra	SVM-8270	1000
SemiVOA Mix 1	Secondary	Ultra	SVM-8271	1000
Custom Standard Mix 3	Secondary	Ultra	CUS-7266	2000
Custom Standard Mix 4	Secondary	Ultra	CUS-7270	2000
Benzidines Mix	Secondary	Ultra	US-105N	2000
Acid Surrogate	-	Restek	31083-510	7500
Base/Neutral Surrogate	-	Restek	31082-510	5000
SV Internal Std Mix	-	Restek	31006	4000

7.2.3 **Primary Calibration Stock Standard** The stock standard is used to prepare the calibration standards. It is stored in a 15 ml Teflon lined cap in a refrigerator at 4°C. The stock standard must be replaced after 6 months or sooner if comparisons with quality control check samples indicate a problem. If any of the primary standards used to prepare the stock standard have expired the stock standard must be discarded. All of the primary standards used to prepare the stock standard should be taken out of the refrigerator and allowed to come to room temperature prior to use - sonicate standard per manufacturer's instructions. Into a 10 ml volumetric flask transfer 2.0 ml each of Mixes 1 & 2 and 1 ml each of Mixes 3 & 4 (7.2.2) together with 267 µL of Acid Surrogate and 400 µL of Base/Neutral surrogate. Volumize to 10.0 ml with Methylene chloride. The volumes of the standards are measured with a 1.0ml Hamilton syringe. This results in a stock solution containing all of the target analytes and surrogates at a concentration of 200 µg/ml. ***Expires in 6 months***.

7.2.4 **Second Source Stock Standard**: The second source stock standard is prepared from the five certified standards from Ultra Scientific (see 7.2.2). 2.0 ml each of Mixes 1 & 2 and 1 ml each of Mixes 3, 4 & 5, & 267 µL of Acid Surrogate and 400 µL of Base/Neutral surrogate is transferred to a 10-ml Class A volumetric and brought to volume with Methylene chloride. The concentration of the SVOA analytes in this solution will be 200 µg/ml. This solution is transferred to a 15-ml vial. The vial is labeled Second Source Semi-VOA Stock Standard with the date prepared, expiration date, concentration, preparer's initials, and the ESS working standard ID number. The standard is stored in the SVOA standard refrigerator. ***Expires in 6 months***.

7.2.5 **Working Standards**:

7.2.5.1 **Tuning Standard**: Ready to use as purchased after transfer to a 1ml vial. Purchased from Supelco, Catalog # 4-7387 at 50 µg/ml.

7.2.5.2 **Calibration Standards**: Eight calibration standards are prepared from the Primary stock standard (7.2.3). All standards are diluted to volume

with Methylene chloride. Standards are stored at 4°C and prepared every six months or sooner if standards present a problem. The continuing calibration level 3 is prepared fresh weekly. One ml of each calibration level is prepared separately in a 2ml vial. Prior to analysis internal standard is added to each of the calibration levels. Using a 25µL syringe, 10µL of the internal standard from Restek (See 7.2.2) is injected into each of the 1.0 ml calibration standards.

Level No.	Standard ID	Volume of Stock	Final Volume	Analyte Conc.	Internal Std. Conc.
1	STD005	25 µL	1.0 ml	5 µg/ml	40 µg/ml
2	STD010	50 µL	1.0 ml	10 µg/ml	40 µg/ml
3	STD025	125 µL	1.0 ml	25 µg/ml	40 µg/ml
4	STD050	250 µL	1.0 ml	50 µg/ml	40 µg/ml
5	STD080	400 µL	1.0 ml	80 µg/ml	40 µg/ml
6	STD120	600 µL	1.0 ml	120 µg/ml	40 µg/ml
7	STD160	800 µL	1.0 ml	160 µg/ml	40 µg/ml
8	STD200	1000 µL	1.0 ml	200 µg/ml	40 µg/ml

7.2.5.3 **Second Source Working Standard**: 250 µL of the second source stock standard (7.2.4) is measured with a 1000µl syringe and diluted to 1000µL in the syringe with Methylene chloride. The final concentration of the analytes in this solution will be 50µg/ml. The solution is transferred to a 2ml-target vial and capped. Using a 25 µL syringe, 10µL of the internal standard from Restek (7.2.2) is injected into the 1.0 ml second source standard. The target vial is labeled as Second Source STD with the working standard ESS lab ID. This standard expires one month after date prepared.

7.2.5.4 **Semi-volatile Surrogate Solution**: This solution contains all acid and base/neutral surrogates. One ml of this solution is spiked into each blank, BS, BSD, sample, MS and MSD ,(for extracts with a final volume of 1ml.). One-half ml (0.5) of this solution is spiked into each blank, BS, BSD, sample, MS and MSD ,(for extracts with a final volume of 0.5ml.).Prepare by obtaining a 5 ml ampoule each of the acid surrogate Base/Neutral surrogate in section 7.2.1. Use a 5 ml class A pipette to transfer 5 ml of each of the ampoules into a 250 ml volumetric. Dilute the surrogate to the 250 ml mark with Acetone. Cap and mix the solution by inverting the volumetric 3 to 5 times. Transfer the solution into a 250 ml amber glass bottle. The bottle is labeled with the working standard ID, name of the solution, concentration, preparer's initials, and expiration date. The expiration date is either the expiration date of the primary standard or six months from date prepared whichever comes first. Before this solution can be used, the concentration of the target analytes must be verified. The laboratory will analyze 1.0ml of this solution like a sample. The result must fall within 15% of the true value.

7.2.5.5 **Semi-volatile Spike Solution**: 5.0 ml each of Mixes 1 & 2 and 2.5 ml each of Mixes 3, 4 & 5 are added to a 50 ml volumetric flask **(section 7.2.2)**. The solution is brought to volume with Acetone. Before the organic prep laboratory can use the spike, the spiking solution is analyzed by taking 1 ml and adding 10 µL internal standard. The percent recoveries are then calculated. If the recoveries are between 80% and 120%, then the lab can use the spiking solution. If not, then a new batch of standard should be used to prepare more of the spiking solution.

8.0 PROCEDURE

8.1 ESS Laboratory's policy is that the audit trail on the Chemstation/Enviroquant software is always on. This ensures that any changes made to the instrument operating method be documented through the audit trail.

8.2 Sample Preparation: Samples must be prepared by one of the following methods:

Matrix	Method	Matrix Summary
Water	3510C	Separatory Funnel (PAH only)
Water	3520C	Liquid-Liquid Extraction
Soil	3541	Automated Soxhlet Extraction
Soil	3550C	Ultrasonic Extraction
Soil	3546	Microwave Extraction

8.3 **Instrument Setup**:

8.3.1 **GC/MS operating conditions**: A detailed description of the instrument operating parameters is in the Enviroquant GC/MS method in Attachment B.

Mass Range	35-500 amu
Scan time	< 1 sec/scan
Initial Temp	40, hold for 2 min
Temp program 1	40-130 at 35/min
Final Temp 1	130, hold for 0.00 min
Temp program 2	130-300 at 12/min
Final temp 2	300, hold for 13 min or until Benzo[g,h,i]perylene elutes
Inj Temp	250 - 275
Transfer line temp	300
Source temp	Manufacturer's specs
Injector	HP EPP (Pulsed pressure program)
Sample volume	1 µl
Carrier Gas	Helium at 1.5 ml/min.

8.3.2 **Mass Spectrometer Evaluation**: Prior to placing an MS into service and after major maintenance, its performance must be evaluated.

8.3.2.1 **Auto Tune**: The instrument is first tuned using the standard Spectra Auto tune from the top menu. See Chapter 3 – "To Interpret the Tune Report" in HP G1034C MS ChemStation User's Guide. This report is printed and placed in the maintenance run log.

8.3.2.2 **Instrument Tune** using HP Software: From the top menu, "Tune MS"⇒"Target Tune"⇒"Tune"⇒"DFTPP Tune". This tunes the MS to preprogrammed targets: Mass 131 32%, Mass 219 32%, and Mass 50 2% with a target peak width of 0.45 amu. The resulting spectra should be close to this target.

8.3.2.3 "Tune Ms" ⇒ "Manual Tune" ⇒ "Load DFTPP.U" ⇒ "Adjparam" can also be used to set the target ions. Check for Air Mass: 28 (Nitrogen), 32 (Oxygen), 18 (Water), and 44 (Carbon Dioxide). All should be less than 10% of the Base 69 Ion. The 218 Ion should be between 30-35% and the 502 Ion should be between 1-2%.

8.3.2.4 Analyst judgment, per instrument history, determines if MS is functioning correctly. Compare prior results to current operating conditions to evaluate the performance of the MS. If the electron multiplier voltage needs to be increased drastically then the mass spec probably needs to be cleaned.

8.4 **Loading the instrument**: All standards and samples are analyzed in 2 ml target vials designed to fit the HP auto-sampler. The target vials are labeled with the ID of the standard or sample using a fine point marker. The tray on the auto-sampler is numbered 1-99. The instrument is set up with one injection system. The Tune and subsequent standards are placed in slots 1-9. This is the standard setup for the calibration standards. Vials can be placed in different slots as long as the slot number is written in the logbook.

Tray # 2	DFTPP Tune Standard
Tray # 3	SVOA Standard Level-1
Tray # 4	SVOA Standard Level-2
Tray # 5	SVOA Standard Level-3
Tray # 6	SVOA Standard Level-4
Tray # 7	SVOA Standard Level-5
Tray # 8	SVOA Standard Level-6
Tray # 9	SVOA Standard Level-7
Tray # 10	SVOA Standard Level-8
Tray # 11	SVOA Second Source Standard Level –4

8.5 **Log Book**: All samples set up on the instrument must be entered into the run logbook. All logbook entries are performed prior to sample analysis. The logbook must be filled out completely as follows: (See Attachment F)

8.5.1 **Date**: include the day, month, and year of analysis.

8.5.2 Vial number: This is a required field.

8.5.3 **Computer file ID**: GC/MS Data is stored in a network directory (Q drive). Data is stored in the appropriate instrument directory (Q:\SVOA\MS3_mf). Each day a batch is set up, a new sub-directory is created with the instrument ID, month, day, and year (Q:\SVOA\MS3_MF\MF022802). Within this directory, data is stored in the following format: A file folder is created for each sample. The ID of each sample file folder has an instrument number followed by a sequential number.

Ex.: SV317823

Where: SV3 is SVOA GC/MS 3
17823 is the 17,823rd file.

8.5.4 **ESS Lab ID:** includes the ID of the standards, samples and all QC samples.

8.5.5 **Analyst's initials**

8.5.6 **Comments**: used for calibration standard IDs, dilution information, and any unusual observations.

8.5.7 **Method 8270/625**: The method in the chromatographic software used to operate and calibrate the instrument. For the 8270C analysis a method could have the ID SV2EA, where: "SV" stands for Semi-Volatile, "2" is the instrument number, and EA is a sequential letter sequence. A copy of a method is in Attachment B.

8.5.8 **Element** Sequence ID and Calibration ID, for soil and water sequences.

8.5.9 RW: Column to indicate samples which have been reviewed.

8.5.10 RPT: Column to indicate runs to be reported.

8.5.11 Imp By: Column to indicate data has been imported into the LIMs system.

8.6 **Initial Calibration**:

8.6.1 A tune check is performed by analyzing a 50ng injection of DFTPP solution described in section 7.2.5.1. The mass spectra of DFTPP ***must*** meet the criteria

in Table 2 prior to standard or sample analysis. Analysis does not begin until all criteria are met. A background subtraction is done when needed to eliminate column bleed or background ions. Do not subtract part of the DFTPP peak.

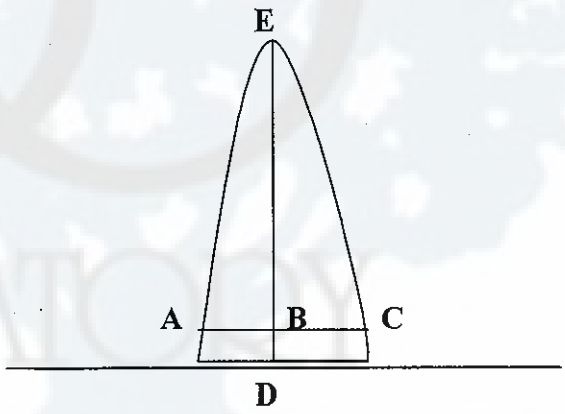
8.6.2 The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. The area count of each of these three analytes is measured. The % Breakdown is calculated as follows:

$$\% \text{ DDT Breakdown} = \frac{\text{Area DDE} + \text{Area DDD}}{\text{Area DDT} + \text{Area DDE} + \text{Area DDD}} \times 100\%$$

8.6.2.1 Benzidine and Pentachlorophenol are also present in the DFTPP mix. There should be no visible peak tailing for either of these compounds. Peak tailing is calculated with the following formula. The tailing factor for pentachlorophenol should not be greater than 5 and Benzidine should not be greater than 3.

Tailing = BC/AB

Peak Height = DE
10% Peak Height = BD
Peak Width at 10% Peak Height = AC
Apex = E



8.6.2.2 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. See daily instrument maintenance. The results of the tune are checked and recorded.

8.6.2.3 If the criteria, set in section 8.6.2.1-8.6.2.2, are not all met, a notation must be made on the data review checklist. When performing analysis for US Army Corps or Navy work, all criteria must be met before analysis begins.

8.6.2.4 All subsequent standards, blanks, samples and spikes associated with the DFTPP analysis must use the same mass spectrometer conditions. The 12 hour tune time starts with the injection time of the DFTPP solution.

8.6.3 10 µL of internal standard solution in section 7.2.1 is spiked into each 1 ml of the calibration standards. In most cases, the base peak ion of each internal standard is used as the primary ion for quantitation. The specific ions used for quantitation of each analyte are listed in the GC EnviroQuant method, (Attachment B). If interferences are present then the next most intense ion is used (For 1,4-Dichlorobenzene-d4 m/z 152 is used for quantitation.) The internal standard solution ID must be entered into the run log page for that run.

8.6.4 After the tune has passed, 1 µL each of the calibration standards is injected and analyzed. Response factors are then calculated for each compound as follows:

RF = (AxCis)/(AisCx)

Where:

Ax = Area of the primary ion for the compound being quantitated
Ais = Area of primary ion for the internal standard
Cis = Concentration of the internal standard (ng/µl)
Cx = Concentration of compound being measured (ng/µl)

8.6.5 The average Response Factor as well as the percent relative standard deviation (%RSD = [SD/RF] x 100) is calculated for each compound. The %RSD should be ≤ 15% for each compound. If this criterion is met, then the relative response factor is considered constant over the calibration range, and the average relative response factor may be used for quantitation. If the %RSD of any compound is greater than 15%, construct calibration curves of area ratio (A/Ais) versus concentration ratio using first or higher order regression fit of the five calibration points. A minimum of five consecutive points are needed for average response or linear regression. A minimum of six consecutive points are needed for quadratic regression. The analyst should select the regression order that introduces the least calibration error into the quantitation. *No more than 15 compounds are to be above 30% RSD.*

8.6.5.1 The %RSD for each individual check compound (Table 3A) ***must*** be less than 30%. If %RSD is greater than 30% clean or replace the injector liner and/or column, then re-calibrate. For each of the calibration compounds the relative retention times should not shift more than 0.06 relative retention time units from the mid-level calibration standard. *Note: For linear and quadratic regression curve, a minimum coefficient of determination of 0.99 is required. The Enviroquant software does not show the COD (R^2) for quadratic analysis. The analyst must enter the calibration into ELEMENT to check the COD on quadratic curves.*

8.6.5.2 The system performance check compounds (SPCC) ***must*** be checked to ensure that the minimum acceptable RFs (0.05) are met before the calibration curve is used. *Note: If the minimum RFs are not met, then*

corrective action must be taken. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at front end of column, and active sites within the chromatographic system.

NOTE: After generating the initial calibration curve in Enviroquant, the analyst must visually check that each calibration standard was entered into the new calibration method. This is accomplished by checking that the area response for one compound or range from each calibration standard's printout corresponds to the area count listed in the calibration method in Enviroquant

8.6.6 After the Initial calibration has been generated, the analyst must create a copy of the Standard 2 file and quantitate the standard with the new calibration. Review of the analyte recoveries in the standard will demonstrate the appropriateness of the calibration curve.

8.6.7 The RRT's are established from the 50ppm mid point standard in the initial calibration.

8.6.8 A second source standard is run after the initial calibration to verify the primary standards. The second source should be between 70-130% (75-125% for DoD/Navy/AFCEE) of the expected concentration, see section 10.0 for extended criteria.

8.7 **Daily GC/MS Calibration**: (Continuing Calibration)

8.7.1 Prior to sample analysis a GC/MS tuning standard must be analyzed. A 50 ng injection of DFTPP must meet the same criteria in Section 8.6.1. This criteria must be met each 12 hour shift. See Table 2. The ID and injection time for the tune standard must be entered into the comment section of the instrument log.

8.7.2 A calibration standard at a mid-calibration range is run immediately after the DFTPP (SSTD050 at 50 ng/µl). The ID for the SSTD050 Standard must be entered into the comment section of the instrument run log. The standard is run each 12 hour shift immediately after the tuning standard. The response factor data is then compared with the average response factor data from the initial calibration. The following formula is used to calculate the percent difference:

$$\% \text{ Difference} = \frac{\overline{RF} - RF}{RF} \times 100\%$$

Where:

$\overline{RF}$ = Average Response factor from the initial calibration.
RF = Response factor from current check standard.

8.7.2.1 Alternatively, if the initial calibration for an analyte consists of a first or higher order curve, the following equations is used to calculate the % drift:

$$\%\text{Drift} = \frac{50 - Cx}{50} \times 100$$

Where:

Cx = Concentration in ng/ul of the analyte

The % Difference/Drift for the CCCs ***must*** be less than 20% for the initial calibration to be assumed valid and the SPCCs must have a minimum RF of 0.05. If the criteria are not met for any one CCC or SPCC, then corrective action must be taken. If no source for the problem can be determined then a new 8-level calibration should be analyzed. All other target analytes should have a % deviation less than 30% (20% for USACE/Navy/DoD/AFCEE). See section 11.0 for corrective action and Tables 3A and 3B for SPCC and CCC compounds.

8.7.3 The internal standard responses and retention times are evaluated in the calibration check. If the retention times shift more than 0.5 minutes from the mid-point calibration standard in the most recent initial calibration, then the GC/MS system must be inspected for possible malfunctions. Also, if the internal standard area shifts more than a factor of two (50 to 200%) from the mid-point calibration standard in the most recent initial calibration, then the GC/MS must be inspected and appropriate corrections made. This usually involves instrument maintenance.

8.7.4 The relative retention times of each compound in each calibration run should agree within 0.06 relative retention time (RRT) units. The RRTs in the continuing calibration standard is compared to the RRTs from the 50ppm std in the ICAL.

8.7.5 A QA/QC summary sheet must be completed recording the results of the tune and the SSTD 050 per each 12-hour period. See Attachment C.

8.7.6 A method blank should be run after the mid-range standard, or at some point in the analytical run to ensure the analytical system is free from contamination.

8.8 **Sample Analysis**:

NOTE: It is not acceptable practice to group QC samples together and/or to analyze QC samples on one instrument and their associated samples on another instrument. Analyst must try to analyze batch QC samples, as capacity allows, along with their associated field samples.

8.8.1 Just prior to sample analysis 10µL of internal standard is spiked into each 1.0 ml of sample extract. The samples are analyzed under the same conditions as the

initial calibration standards. The internal standard identification must be recorded in the run log.

8.8.2 Samples must be analyzed within the 12-hour tune time, which begins at the injection time of the tune standard. Any other analysis run after the 12-hour tune time, i.e., 625, Siloxanes and Pesticides must be identified as such.

8.8.3 Sample dilution is made when the response of any quantitation ion exceeds the initial calibration curve. Additional internal standard is always added to the diluted extract to maintain the required 40 ng/µL concentration of each internal standard. Dilutions are also made when samples will not inject into the instrument due to their viscosity.

8.9 **Qualitative analysis**:

8.9.1 **Target Compound List**: An analyte is identified by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound. The standard mass spectra are obtained from the SSTD050 (Level 3) of the initial calibration. Two criteria must be satisfied to verify identification:

8.9.1.1 The sample component must elute at the same relative retention time as the standard component. It must elute within ±0.06 RRT units of the RRT of the standard. The RRT of the sample component is compared to the RRT from the 50ppm std in the ICAL.

8.9.1.2 The sample and standard mass spectra should correspond. All ions greater than 10% in the standard mass spectrum must be present in the sample mass spectrum. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Ex. An ion with a relative ratio of 50% in the standard must be between 20 and 80% in the sample.)

8.9.2 **Tentatively Identified Compounds**: For samples containing components not associated with the calibration standard, a library search can be made for tentative identification. Guidelines for tentative identification are as follows:

8.9.2.1 Relative intensities of all major ions greater than 10% should be present in the sample spectrum.

8.9.2.2 The relative intensity of major ions should agree within plus or minus 20%. (Ex. An ion with a relative ratio of 50% in the standard must be between 30 and 70% in the sample.)

8.9.2.3 Molecular ions present in the reference spectra should also be in the sample spectrum.

8.9.2.4 Ions not present in the reference spectra should be reviewed for possible background contamination. Background subtractions should be attempted to account for this.

9.0 CALCULATIONS (DATA INTERPRETATION)

9.1 **Quantitative analysis**: When a compound is identified the quantitation of that compound is based on the integrated abundance of the primary ion. Quantitation is done with an internal standard. The internal standard used is the one nearest the retention time of the compound of interest.

9.1.1 **Water quantitation**:

$$\text{Concentration (ug/L)} = \frac{(Ax)(Is)(Vt)(Dil)}{(Ais)(RF_{avg})(Vo)Vi}$$

Where:

Ax	=	Area of primary ion for compound being measured
Is	=	Amount of internal standard injected (ng)
Vt	=	Volume of total extract (µL)
Ais	=	Area of primary ion of internal standard
RF_{avg}	=	Response factor of compound being measured
Vo	=	Volume of water extracted (ml)
Vi	=	Volume of extract injected (µL)
Dil	=	Dilution factor

9.1.2 **Sediment/ Soil Sludge (on dry weight basis) and waste (on wet weight basis):**

$$\text{Concentration (µg/Kg)} = \frac{(Ax)(Is)(Vt)(Dil)}{(Ais)(RF_{avg})(Vi)(Ws)(D)}$$

Where:

Ax, Is, Vt, Dil, Ais, RF_{avg}, and Vi are the same as in 9.1.1
Ws = Weight of sample extracted (grams)
D = % dry weight of sample. (1 for wet weight basis)

9.1.3 **Quantitation of Tentatively Identified Compounds**: Quantitation of a TIC is done assuming a response factor of one. The formulas above are used with the following modifications:

Ax = Area of the total ion chromatogram for compound being measured
Ais = Area of the total ion chromatogram for the internal standard

10.0 QUALITY ASSURANCE/QUALITY CONTROL

10.1 The laboratory operates a quality control program to demonstrate the laboratory's capability and, with an ongoing analysis of spiked samples, to document the quality of the data generated. The ongoing data quality checks are compared with established

performance criteria to determine if the result of the analysis meets the performance criteria of the method.

10.2 **Accuracy and Precision** All laboratory personnel must demonstrate initial proficiency for each sample preparation method/matrix that he or she performs. All new employees must successfully demonstrate initial proficiency prior to independently performing analysis on real samples. This must be accomplished by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The initial proficiency results will become part of each employee's training file.

QC Sample Preparation:
Spiking Solution: Four QC samples must be prepared from a spiking solution with the analytes of interest. The spiking solution should be made using standards **prepared independently from those used for calibration**. The samples must be prepared at a concentration that would result in data falling within the middle of the calibration curve, 50 µg/L. In most cases the blank spike or matrix spike solution is used. Prep: The samples are prepared in a clean matrix. In most cases this initial demonstration will simply be a matter of preparing four blank spikes with a batch of samples.

Sample Analysis:
The four QC samples must be analyzed within the criteria of the method being evaluated. The QC samples must be handled in exactly the same manner as actual samples.

Accuracy Calculation:
Accuracy is defined as the closeness of agreement between an observed value and an accepted reference value. Each of the four spiked samples will be calculated for percent recovery. The average of the percent recovery values is the accuracy result.

Precision Calculation:
Precision is defined as the agreement of a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by the relative standard deviation (RSD) of the four QC samples.

$$\%RSD = (s / \bar{x})\ 100\ \%$$

Where:

s = Standard Deviation of a finite number of values. On a scientific calculator use the σ**xn-1** key.

$\bar{x}$ = The average of the four QC sample % recoveries.

Reporting Accuracy and Precision Accuracy and Precision data should be presented with the following minimum info:

Matrix: Prep Method:
Clean-up Method: Analysis Method:

Date Extracted: Date Analyzed:
Sample Prepared by: If Applicable Sample Fractionated by: If Applicable
Sample Analyzed by: Accuracy:
Precision:

Parameter	*% Rec. QC 1*	*% Rec. QC 2*	*% Rec. QC 3*	*% Rec. QC 4*	*Average Recovery*	*Standard Deviation*	*%RSD*

Interpretation of Results:
The acceptance range for Accuracy is 45-135% with <30% RSD, 4 sporadic marginal exceedances are allowed. If any of the accuracy and precision results do not fall within the criteria then re-prep and reanalyze all QC samples only for those analytes outside criteria.

10.3 Method blanks are run with each batch to demonstrate that interferences from the analytical system, glassware, and reagents are under control. The blanks are carried through all stages of sample preparation. All target analytes should be less than ½ the MRL for the blank. See section 11.0 for corrective action for out of criteria results.

10.4 Perform DFTPP tune every 12 hours. Tuning acceptance criteria are presented in Table 2. The computer software will evaluate the tune information. The analyst should be aware of the process used. The tune *must* meet these criteria before sample analysis begins.

10.5 Run an initial calibration curve, using the primary source standards, each time major instrument maintenance occurs or if the CCV does not meet acceptance criteria. Acceptance criteria are presented in Section 8.6.

10.6 Run initial calibration verification (ICV) standard using secondary standards (7.2.5.3) following the initial calibration curve. Acceptance criteria are listed in Section 8.6.6. If the ICV does not meet criteria, re-analyze. If the second consecutive ICV does not meet criteria, then perform system maintenance and re-calibrate the system. The laboratory is allowed up to 5 sporadic marginal exceedances with the expanded criteria of 50-150%. ***NOTE: When analyzing samples for DoD/Navy/AFCEE/USACE no sporadic marginal exceedances are allowed***

10.7 Run mid-point Continuing Calibration Verification (CCV) at 50 µg/L using the primary source standards on a daily basis before sample analysis. Also run a CCV every 12 hours during an analytical sequence. Acceptance criteria are listed in Section 8.7. See section 11.0 for corrective action for out of criteria results.

10.8 A blank spike and blank spike duplicate must be prepared and analyzed with each batch of samples. BS/BSDs are prepared using the second source standard and contain all target analytes. Control limits are 40-140% for the base/neutral compounds (45-135% for USACE) and 30-130% for the acid compounds (35-140% for USACE). See Table 4 for DoD/Navy/AFCEE control limits. The BS/BSD should have a %RPD of ≤ 20% for waters and ≤ 30% for soils. If > 20% of all compounds fail or if >15% of any one

category fails then re-extraction of the batch is necessary. Otherwise, note exceptions in the project narrative. ***DoD/Navy/AFCEE/USACE allow Sporadic Marginal Exceedances for BS/BSD as listed in the below chart. For the USACE (Shell), sporadic marginal exceedances have the expanded criterion of*** *15-150% for waters and 25-150% for soils.* ***See Table 4 for DoD and AFCEE blank spike acceptance criteria.***

Number of Analytes in BS	Allowable number of Marginal Exceedances
>90	5
71-90	4
51-70	3
31-50	2
11-30	1
<11	0

Exceedances should be within marginal exceedance limits (Table 4) or expanded criterion listed above.

10.8.1 Control charts will be maintained for the BS/BSD for a representative subset of target analytes and surrogate spikes. Annually, control limits will be determined for all target analytes and surrogates for comparison to default limits.

10.9 On an ongoing basis the laboratory analyzes matrix spikes and matrix spike duplicates from each batch of 20 samples. Matrix spike control limits are derived from BS/BSD samples, see section 10.8. For samples outside control limits, see Section 11.0 for corrective action.

10.10 Surrogates are added to all samples and QC samples. If the surrogate recoveries for customer or QC samples are outside control limits, see Section 11.0 for corrective action. Control limits for DoD/Navy/AFCEE surrogate are listed in Table 4.

Matrix	B/N Analytes	Acid Analytes
Soil	30-130% (USACE 45-135%)	30-130% (USACE 35-140%)
Water	30-130% (USACE 45-135%)	15-110% (USACE 35-140%)

10.11 The relative retention times (RRTs) need to be checked for each identified compound in samples, and compared to standard RRT. Acceptance criteria are presented in Section 11.0.

10.12 Internal standard area counts for standards and samples must meet specifications as described in Section 11.10.

10.13 Data shall be checked to ascertain if it conforms to accepted practices. All sample analytical results used for final data reporting must be between the low standard and the high standard. Results, which fall below the low standard or above the high standard, are to be reported as estimated values. Corrective actions are described in Section 11.0.

10.14 MDLs are determined annually in reagent water or organic-free sand/sodium sulfate and verified by an MDL check. See SOP 110_0013 for MDL specification. (Project-specific requirements may require that the MDL study be performed in the site-specific matrix.).

10.15 All manual integrations must be printed, when made, for verification. Refer to manual integration policy in SOP 110_0016.

11.0 DATA VALIDATION

The items shall be verified and documented using the data review checklist in Attachment E.

11.1 Ensure that the DFTPP tune was run at the beginning of each 12-hour sequence for each batch of samples analyzed. The acceptance criteria are listed in Table 2.

11.1.1 If the DFTPP acceptance criteria are not met, perform any or all of the following corrective actions:

Re-inject DFTPP.
Retune with PFTBA, and then re-inject DFTPP.
Clean MS source, retune with PFTBA, and re-inject DFTPP.

11.1.2 If the tuning criteria still cannot be met after performing the above, have the mass spectrometer serviced by manufacturer representative.

11.2 After an initial calibration curve is analyzed, ensure that the following criteria were met.

11.2.1 If the %RSD of any method target analyte is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor may be used for quantitation.

11.2.2 If the %RSD of any method target analyte is greater than 15%, calibration curves must be constructed using first or higher order regression fits. A minimum of 5 points must be used for linear regression and six points must be used for Quadratic Regression. The corresponding coefficient of determination (R^2) must be 0.99 or greater.

11.2.3 Evaluate CCCs and SPCCs for the criteria listed in section 8.6. CCCs and SPCCs ***must*** meet criteria no matter what calibration option is used.

11.2.4 For the remaining compounds if greater than 20% of the compounds (15 for full list) have %RSD > 30, then recalibration is required.

11.2.5 If the acceptance criteria are not met, then the following corrective actions should be performed: (1) adjust the instrument and/or perform instrument maintenance; or (2) narrow the calibration range using six standards at different concentrations. The low end of the calibration curves must be carefully watched.

11.3 After an initial calibration (minimum of 5 point for average RF or linear regression, minimum of 6 points for quadratic regression) is performed, analyze initial calibration verification (ICV) standard. The percent difference must be within ±30% (±25% for DoD/Navy/AFCEE) for all target analytes with allowance for a maximum of ***5 sporadic marginal exceedances (No allowances for DoD/Navy/USACE/AFCEE)***. Sporadic marginal exceedances have the expanded criterion of 50-150% drift. If criteria are not met, reanalyze the ICV or prepare a new calibration curve as necessary.

11.4 After the continuing calibration verification (CCV) standard is analyzed, ensure it was run at the required frequency (every 12 hours or initially before daily analysis). In addition, the following acceptance criteria must be met.

11.4.1 System Performance Check Compounds (SPCCs): This is the same check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins.

11.4.2 Calibration Check Compounds (CCCs) listed in Table 3A are evaluated to check the validity of the initial calibration with a limit of ≤20% drift or difference, ≤ 30% drift for all other method target analytes (allow up to 20% of non-CCC compounds >30%D or up to 15% in each category). ***NOTE: For DoD/Navy/AFCEE/USACE samples, all compounds must have ≤20% drift, no allowances. DoD allows the use of a grand mean where the average of all compounds must be within ±20%, with no individual analytes (except CCCs) >25%.***

11.4.2.1 If these criteria are exceeded, corrective action is necessary. If corrective action fails to produce consecutive (immediate) calibration verification within acceptance criteria, then the analyst may perform maintenance to try to troubleshoot the instrument. After maintenance, the analyst must analyze two consecutive CCV at two concentration (one at or below mid-range) to demonstrate that corrective action was successful. If not, then a new initial calibration must be generated. These criteria must be met before sample analysis begins and re-analysis of samples up to the last acceptable CCV standard must occur with the following exceptions:

- When the acceptance criteria is exceeded high, high bias, then all samples that are non-detects may be reported.

- When the acceptance criteria for the CCV are exceeded low, low bias, sample results that exceed a maximum regulatory limit may be reported. **NOTE: When analyzing samples for USACE/DoD/Navy/AFCEE, all samples associated with a CCV below lower control limit must be re-analyzed.**

11.4.3 When performing analysis for MA MCP, Navy, USACE (Shell), DoD, or

AFCEE any analyte outside of criteria in the CCV must be noted in the project narrative

11.5 Assess the method blank. The analyst must confirm that the method blank was analyzed at the required frequency and all target analytes are below the MRL (½ the MRL for DoD/Navy/AFCEE/USACE). Analytical batches with Method blanks outside acceptance criteria will be re-prepped and re-analyzed (where sample volume permits) with the following exceptions:

11.5.1 Samples that are at least twenty times higher than the method blank may be reported.

11.5.2 When the method blank is less than 5% of the regulatory limit associated with the analyte the method blank would be acceptable.

11.5.3 If the analyte is found in the method blank above the MRL (½ the MRL for DoD/Navy/AFCEE/USACE) but is not in any of the associated samples, no corrective action is needed.

11.5.4 Any results that are reported with method blank contamination must be B-flagged.

11.6 Assess that the matrix spike/matrix spike duplicates were analyzed at the required frequency. Acceptance criteria are listed in 10.9. If criteria are not met, perform the following corrective actions as appropriate.

11.6.1 If both BS/BSD and MS/MSD recoveries are unacceptable, then the entire batch of field and QC samples must be reanalyzed.

11.6.2 If the MS/MSD is unacceptable, but the BS/BSD is acceptable, then a potential matrix effect has been identified. Reasonable attempts must be made to address matrix interference. The client must be notified in the case narrative of the matrix problem.

11.7 Check the surrogate calculations for correctness for all samples, blanks, BS/BSD, MS, and MSD. The following acceptance criteria apply to surrogate recoveries.

11.7.1 The surrogate recoveries for all QC samples must be within control limits. If the BS/BSD and/or method blank recoveries are outside limits, re-analysis must be performed for verification. If still outside limits, then corrective action is to re-extract the batch.

11.7.2 If sample surrogate recoveries are outside control limits (10.10), the sample should be re-analyzed. If the results are still outside the limits, then re-prep and re-analyze the sample(s). An exception to this criterion is surrogates diluted out of sample due to matrix problems. In addition, if an obvious interference is present, the laboratory does not have to re-extract the sample but must provide

the chromatogram with the project. *ESS Laboratory's policy allows for one acid and one base surrogate to be outside control limits before re-prepping sample batches (no allowance for DoD/Navy/AFCEE/USACE samples; apply J-flag for specific analytes in all field samples collected from the same site matrix as the parent if acceptance criteria are not met; apply Q-flag to specific analytes in all QC samples in the associated preparatory batch). If any surrogate is<10%, then sample must be re-prepped.*

11.8 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.9 The relative retention times must be checked for all identified compounds in both standards and samples. The internal standard absolute retention times must also be checked for all analyses. Acceptance criteria are as follows:

11.9.1 The relative retention times of each compound in each calibration and sample run should agree within 0.06 relative retention time (RRT) units. Late-eluting compounds usually have much better agreement.

11.9.2 Internal standard retention time:

11.9.2.1 If the retention time for any CCV internal standard changes by more than 30 seconds from the **mid-point standard of the initial calibration**, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.9.3 If the retention time for any sample internal standard changes by more than 30 seconds from the daily calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required.

11.10 The analyst must verify that ion abundance meets specific criterion for the various analyses. The following acceptance criteria shall be checked for all appropriate samples.

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

11.10.2 The relative intensities of ions must agree within plus or minus 30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample abundance must be between 20 and 80 percent.)

11.10.2.1 Molecular ions present in the reference spectrum should be present in the sample spectrum.

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

11.10.2.3 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.10.3For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. When serving the role as QA (or referee) laboratory, tentatively identified compounds (TICs) are always reported. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. For example, the RCRA permit or waste de-listing requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches will the mass spectral interpretation specialist assign a tentative identification. Guidelines for making tentative identification are as follows:

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

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

11.10.3.3 Molecular ions present in the reference spectrum should be present in the sample spectrum. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

11.10.3.4 Ions present in the reference spectrum, but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.11 The analyst will check the internal standard area counts for all calibration standards, QC samples, and samples for quantitation. If the area for any of the CCV internal standards changes by a factor of two (-50% to +100%) from the mid-point standard of the initial calibration, the mass spectrometer must be inspected for malfunctions and corrections

must be made, as appropriate. All sample internal standards must be -50 to 200% of their associated CCV. If the internal standard area counts fail this criterion, the following corrective actions should be considered:

11.11.1 Check to ensure there was no error in internal standards preparation or addition. Also, check instrument performance.

11.11.2 If the CCV failed criterion, re-analyze once. If this second consecutive CCV still does not meet criterion, then a new initial calibration must be performed.

11.11.3 If the internal standards for samples are outside criterion, then re-analyze once. If re-analysis produces results within criteria, then report these results. If the re-analysis is still outside criterion, discuss matrix issue in project narrative. ***NOTE: If an obvious matrix effect is displayed on the chromatogram (Unresolved complex mixture, UCM), then re-analysis is not necessary. MA-MCP projects must have a copy of the chromatogram included with the report.***

11.12 The analyst must verify all reported results are derived from analytical results that are below the highest standard of the initial calibration curve and above the low standard. Values reported below the low standard are to be reported as estimated values (J values). For samples that exceed the calibration curve, dilute and analyze an appropriate sample aliquot.

11.13 Besides the items listed in Sections 11.1 through 11.11, the analyst should also verify the additional items as noted in Attachment E. A second level of review must be performed by a second analyst; results are recorded on Attachment E.

12.0 REFERENCES

12.1 Method 8270C, Test Methods for the Analysis of Solid Waste, Third Edition.
12.2 HP GC EnviroQuant User's Guide, HPG1045A
12.3 HP Environmental Data Analysis User's Guide HPG0032C
12.4 HP 6890 GC Operations Manual
12.5 HP 5890 Series II Operations Manual
12.6 HP 5971A MS Operations Manual
12.7 HP 5973 MS Operations Manual
12.8 NELAC Quality Systems Chapter 5, Version 16, July 2002
12.9 DoD Quality Systems Manual, Final Version 3, January 2006

13.0 POLLUTION PREVENTION and WASTE MANAGEMENT

13.1 ESS Laboratory's policies on pollution prevention and waste management are covered in SOP 90_0002, Hazardous Waste Contingency and Emergency Response Plan. All employees are trained in the requirements of the SOP.

14.0 METHOD PERFORMANCE

14.1 Precision and Accuracy data must be generated by all employees before performing this analysis on client samples. The data is generated by analyzing a method blank and four blank spike samples. Acceptance criteria are 45-135% Recovery and %RSD of ≤ 30%. Four (4) sporadic marginal exceedances are allowed.

14.2 The precision and accuracy data in Table 1 were obtained using the SOP. Values are in ug/L.

15.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Table 1. Typical Precision and Accuracy data generated 1/3/2005

Compound Name	Spk Amt	Avg	RSD	%Rec	Compound Name	Spk Amt	Avg	RSD	%Rec
N-Nitrosodimethylamine	100	62.13	9	62.1	Acenaphthylene	100	64.46	5	64.5
Pyridine	100	33.39	55	33.4	2,6-Dinitrotoluene	100	85.33	11	85.3
2-Fluorophenol (Surr)	150	83.27	7	55.5	2-Nitroaniline	100	79.96	9	80.0
bis(2-Chloroethyl)ether	100	66.28	7	66.3	Acenaphthene	100	77.24	6	77.2
Phenol-d5 (Surr)	150	85.70	6	57.1	2,4-Dinitrophenol	100	49.15	20	49.1
2-Chlorophenol	100	66.38	6	66.4	Dibenzofuran	100	78.48	7	78.5
Phenol	100	61.65	5	61.6	4-Nitrophenol	100	80.25	10	80.2
Aniline	100	77.02	31	77.0	3-Nitroaniline	100	54.80	55	54.8
2-Chlorophenol-d4(Surr)	150	92.25	7	61.5	2,4-Dinitrotoluene	100	85.21	11	85.2
1,3-Dichlorobenzene	100	65.12	6	65.1	Fluorene	100	81.66	6	81.7
1,4-Dichlorobenzene	100	66.55	4	66.5	2,3,4,6-Tetrachlorophenol	100	88.20	8	88.2
1,2 Dichlorobenzene-d4(Surr)	100	69.15	7	69.2	Diethylphthalate	100	82.05	7	82.1
1,2-Dichlorobenzene	100	69.31	4	69.3	4-Chloro-phenyl-phenyl ether	100	79.66	6	79.7
Benzyl Alcohol	100	78.72	7	78.7	4-Nitroaniline	100	68.82	21	68.8
bis(2-chloroisopropyl)ether	100	64.68	6	64.7	4,6-Dinitro-2-methylphenol	100	74.79	17	74.8
2-Methylphenol	100	66.15	7	66.1	n-Nitrosodiphenylamine	100	58.19	12	58.2
Acetophenone	100	75.74	6	75.7	Azobenzene	100	74.11	7	74.1
n-Nitroso-di-n-propylamine	100	74.40	7	74.4	2,4,6-Tribromophenol (Surr)	100	120.65	6	120.7
Hexachloroethane	100	68.22	6	68.2	4-Bromophenyl-phenylether	100	81.95	7	81.9
3+4-Methylphenol	100	69.03	7	69.0	Hexachlorobenzene	150	82.06	7	54.7
Nitrobenzene-d5 (Surr)	100	69.26	12	69.3	Pentachlorophenol	100	84.10	9	84.1
Nitrobenzene	100	67.77	11	67.8	Phenanthrene	100	77.83	7	77.8
Isophorone	100	68.67	11	68.7	Anthracene	100	76.01	7	76.0
2-Nitrophenol	100	71.52	10	71.5	Carbazole	100	71.52	8	71.5
Benzoic Acid	100	27.79	76	27.8	Di-n-butylphthalate	100	78.48	8	78.5
2,4-Dimethylphenol	100	56.93	26	56.9	Fluoranthene	100	82.08	8	82.1

bis(2-Chloroethoxy)methane	100	63.50	7	63.5	Benzidine	100	14.07	NA	NA
2,4-Dichlorophenol	100	72.11	11	72.1	Pyrene	100	75.55	7	75.6
1,2,4-Trichlorobenzene	100	68.79	10	68.8	Terphenyl-d14 (Surr)	100	80.18	7	80.2
Naphthalene	100	66.01	10	66.0	Butylbenzylphthalate	100	77.04	8	77.0
4-Chloroaniline	100	49.45	28	49.4	3,3'-Dichlorobenzidine	100	40.93	67	40.9
Hexachlorobutadiene	100	71.25	9	71.2	Benzo(a)anthracene	100	76.79	7	76.8
4-Chloro-3-methylphenol	100	73.73	11	73.7	Chrysene	100	77.87	8	77.9
2-Methylnaphthalene	100	67.72	11	67.7	bis(2-Ethylhexyl)phthalate	100	77.70	7	77.7
Hexachlorocyclopentadiene	100	8.96	16	9.0	Di-n-octylphthalate	100	86.23	7	86.2
2,4,6-Trichlorophenol	100	79.95	7	79.9	Benzo(b)fluoranthene	100	69.76	14	69.8
2,4,5-Trichlorophenol	100	82.78	6	82.8	Benzo(k)fluoranthene	100	121.30	21	121.3
2-Fluorobiphenyl (Surr)	100	78.33	8	78.3	Benzo(a)pyrene	100	75.50	8	75.5
Biphenyl	100	77.41	7	77.4	Indeno(1,2,3-cd)pyrene	100	88.02	14	88.0
2-Chloronaphthalene	100	78.08	6	78.1	Dibenzo(a,h)anthracene	100	83.84	12	83.8
Dimethylphthalate	100	79.39	8	79.4	Benzo(g,h,i)perylene	100	93.13	17	93.1

TABLE 2
DFTPP KEY IONS AND ION CRITERIA

MASS	ION ABUNDANCE CRITERIA	MASS	ION ABUNDANCE CRITERIA
51	30% to 60% of mass 198	199	5% to 9% of mass 198
68	< 2% of mass 69	275	10% to 30% of mass 198
70	< 2% of mass 69	365	> 1% of mass 198
127	40% to 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% to 23% of mass 442

Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and Pentachlorophenol should be present at their normal response and no peak tailing should be visible.

Table 3A
Calibration Check Compounds (CCC)

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol

1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

Table 3B
System Performance Check Compounds (SPCC)

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

Table 4

DoD Quality Systems Manual and AFCEE QAAP Blank Spike QC Limits

	DoD				AFCEE			
AQUEOUS								
Analyte	LCL	UCL	LMEL	UMEL	LCL	UCL	LMEL	UMEL
1,2,4-Trichlorobenzene	35	105	25	120	37	120	25	120
1,2-Dichlorobenzene	35	100	20	115	33	120	20	120
1,2-Diphenylhdrazine	55	115	45	120	-	-	-	-
1,3-Dichlorobenzene	30	100	20	110	32	120	20	120
1,4-Dichlorobenzene	30	100	20	110	32	120	20	120
2,4,5-Trichlorophenol	50	110	40	120	49	120	40	120
2,4,6-Trichlorophenol	50	115	40	125	49	126	40	125
2,4-Dichlorophenol	50	105	40	115	48	120	40	120
2,4-Dimethylphenol	30	110	15	125	28	120	15	125
2,4-Dinitrophenol	15	140	10	160	25	130	10	160
2,4-Dinitrotoluene	50	120	40	130	51	120	40	130
2,6-Dinitrotoluene	50	115	35	130	49	120	35	130
2-Chloronaphthalene	50	105	40	115	49	120	40	120
2-Chlorophenol	35	105	25	115	37	120	25	120
2-Methylnaphthalene	45	105	35	115	45	120	35	120
2-Methylphenol	40	110	25	120	38	120	25	120

2-Nitroaniline	50	115	35	125	48	120	35	125
2-Nitrophenol	40	115	25	125	39	123	25	125
3,3'-Dichlorobenzidine	20	110	10	125	20	120	10	125
3+4-Methylphenol	30	110	20	125	32	120	20	125
3-Nitroaniline	20	125	10	145	20	125	10	145
4,6-Dinitro-2-methylphenol	40	130	25	145	40	130	25	145
4-Bromophenyl-phenylether	50	115	40	125	52	120	40	125
4-Chloro-3-methylphenol	45	110	35	120	47	120	35	120
4-Chloroaniline	15	110	10	125	20	120	10	125
4-Chloro-phenyl-phenyl ether	50	110	40	120	50	120	40	120
4-Nitroaniline	35	120	20	130	36	120	20	130
4-Nitrophenol	0	125	0	145	20	120	0	145
Acenaphthene	45	110	35	120	47	120	35	120
Acenaphthylene	50	105	40	115	50	120	40	120
Anthracene	55	110	45	120	54	120	45	120
Benzo(a)anthracene	55	110	45	120	56	120	45	120
Benzo(a)pyrene	55	110	45	120	53	120	45	120
Benzo(b)fluoranthene	45	120	35	130	45	124	35	130
Benzo(g,h,i)perylene	40	125	25	135	38	123	25	135
Benzo(k)fluoranthene	45	125	30	135	45	124	30	125
Benzoic Acid	0	125	0	150	20	120	0	150
Benzyl Alcohol	30	110	15	125	30	120	15	125
bis(2-Chloroethoxy)methane	45	105	35	115	46	120	35	120
bis(2-Chloroethyl)ether	35	110	25	120	37	120	25	120
bis(2-chloroisopropyl)ether	25	130	10	150	26	131	10	150
bis(2-Ethylhexyl)phthalate	40	125	30	140	42	126	30	140
Butylbenzylphthalate	45	115	35	130	46	120	35	130
Carbazole	50	115	35	130	-	-	-	-
Chrysene	55	110	45	120	55	120	45	120
Dibenzo(a,h)anthracene	40	125	30	140	42	127	30	140
Dibenzofuran	55	105	45	115	54	120	45	120
Diethylphthalate	40	120	30	130	41	120	30	130
Dimethylphthalate	25	125	10	145	25	127	10	145
Di-n-butylphthalate	55	115	45	125	54	120	45	125
Di-n-octylphthalate	35	135	20	155	37	137	20	155
Fluoranthene	55	115	45	125	54	120	45	125
Fluorene	50	110	40	120	50	120	40	120
Hexachlorobenzene	50	110	40	120	52	120	40	120
Hexachlorobutadiene	25	105	15	115	27	120	15	120
Hexachloroethane	30	95	15	105	28	120	15	120
Indeno(1,2,3-cd)pyrene	45	125	30	140	43	125	30	140
Isophorone	50	110	40	125	50	120	40	125
Naphthalene	40	100	30	115	39	120	30	120

Nitrobenzene	45	110	35	120	44	120	35	120
N-Nitrosodimethylamine	25	110	10	125	-	-	-	-
n-Nitroso-di-n-propylamine	35	130	20	145	34	128	20	145
n-Nitrosodiphenylamine	50	110	35	120	48	120	35	120
Pentachlorophenol	40	115	25	130	38	120	25	130
Phenanthrene	50	115	40	130	51	120	40	130
Phenol	0	115	0	135	20	120	0	135
Pyrene	50	130	35	140	49	128	35	140
Surrogates:								
2-Fluorobiphenyl	50	110	-	-	48	120	-	-
Terphenyl-d14	50	135	-	-	51	135	-	-
2,4,6-Tribromophenol	40	125	-	-	42	124	-	-
2-Fluorophenol	20	110	-	-	20	120	-	-
Phenol-d5/d6	10	115	0	135	20	120	-	-
Nitrobenzene-d5	40	110	-	-	41	120	-	-
SOIL								
Analyte	LCL	UCL	LMEL	UMEL	LCL	UCL	LMEL	UMEL
1,2,4-Trichlorobenzene	45	110	30	120	44	125	30	125
1,2-Dichlorobenzene	45	95	35	105	45	125	35	125
1,2-Diphenylhdrazine								
1,3-Dichlorobenzene	40	100	30	110	39	125	30	125
1,4-Dichlorobenzene	35	105	25	115	35	125	25	125
2,4,5-Trichlorophenol	50	110	40	120	49	125	40	125
2,4,6-Trichlorophenol	45	110	30	120	43	125	30	125
2,4-Dichlorophenol	45	110	35	120	45	125	35	125
2,4-Dimethylphenol	30	105	20	115	32	125	20	125
2,4-Dinitrophenol	15	130	10	150	25	132	10	150
2,4-Dinitrotoluene	50	115	35	130	48	125	35	130
2,6-Dinitrotoluene	50	110	35	125	48	125	35	125
2-Chloronaphthalene	45	105	35	115	45	125	35	125
2-Chlorophenol	45	105	35	115	44	125	35	125
2-Methylnaphthalene	45	105	35	115	47	125	35	125
2-Methylphenol	40	105	30	115	40	125	30	125
2-Nitroaniline	45	120	30	130	44	125	30	130
2-Nitrophenol	40	110	30	120	42	125	30	125
3,3'-Dichlorobenzidine	10	130	0	145	25	128	0	145
3+4-Methylphenol	40	105	30	120	41	125	30	125
3-Nitroaniline	25	110	15	125	27	125	15	125
4,6-Dinitro-2-methylphenol	30	135	10	155	29	137	10	155
4-Bromophenyl-phenylether	45	115	35	130	46	125	35	130
4-Chloro-3-methylphenol	45	115	35	125	46	125	35	125
4-Chloroaniline	10	95	0	110	25	125	0	125
4-Chloro-phenyl-phenyl ether	45	110	35	120	47	125	35	125

4-Nitroaniline	35	115	20	125	34	125	20	125
4-Nitrophenol	15	140	10	160	25	138	10	160
Acenaphthene	45	110	35	120	46	125	35	125
Acenaphthylene	45	105	35	115	44	125	35	125
Anthracene	55	105	45	115	53	125	45	125
Benzo(a)anthracene	50	110	40	120	52	125	40	125
Benzo(a)pyrene	50	110	40	120	50	125	40	125
Benzo(b)fluoranthene	45	115	35	125	45	125	35	125
Benzo(g,h,i)perylene	40	125	25	140	38	126	25	140
Benzo(k)fluoranthene	45	125	30	135	45	125	30	135
Benzoic Acid	0	110	0	130	25	125	0	130
Benzyl Alcohol	20	125	10	140	25	125	10	140
bis(2-Chloroethoxy)methane	45	110	30	120	43	125	30	125
bis(2-Chloroethyl)ether	40	105	25	115	38	125	25	125
bis(2-chloroisopropyl)ether	20	115	10	130	25	125	10	130
bis(2-Ethylhexyl)phthalate	45	125	35	140	47	127	35	140
Butylbenzylphthalate	50	125	35	135	49	125	35	135
Carbazole	45	115	30	130	-	-	-	-
Chrysene	55	110	45	120	43	125	45	125
Dibenzo(a,h)anthracene	40	125	25	140	41	125	25	140
Dibenzofuran	50	105	40	110	51	125	40	125
Diethylphthalate	50	115	40	125	50	125	40	125
Dimethylphthalate	50	110	40	120	49	125	40	125
Di-n-butylphthalate	55	110	45	120	56	125	45	125
Di-n-octylphthalate	40	130	25	145	41	132	25	145
Fluoranthene	55	115	45	125	54	125	45	125
Fluorene	50	110	40	115	49	125	40	125
Hexachlorobenzene	45	120	35	130	47	125	35	130
Hexachlorobutadiene	40	115	25	130	40	125	25	130
Hexachloroethane	35	110	20	120	34	125	20	125
Indeno(1,2,3-cd)pyrene	40	120	25	135	38	125	25	135
Isophorone	45	110	30	125	43	125	30	125
Naphthalene	40	105	30	120	40	125	30	125
Nitrobenzene	40	115	30	125	41	125	30	125
N-Nitrosodimethylamine	20	115	10	130	-	-	-	-
n-Nitroso-di-n-propylamine	40	115	30	125	40	125	30	125
n-Nitrosodiphenylamine	50	115	40	125	49	125	40	125
Pentachlorophenol	25	120	10	135	25	125	10	135
Phenanthrene	50	110	40	120	50	125	40	125
Phenol	40	100	30	110	39	125	30	125
Pyrene	45	125	35	135	46	125	35	135
Surrogates:								
2-Fluorobiphenyl	45	105	-	-	43	120	-	-

Terphenyl-d14	30	125	-	-	32	120	-	-
2,4,6-Tribromophenol	35	125	-	-	36	126	-	-
2-Fluorophenol	35	105	-	-	37	120	-	-
Phenol-d5/d6	40	100	-	-	40	120	-	-
Nitrobenzene-d5	35	100	-	-	37	120	-	-

%RPD for DoD is ≤30%. %RPD for AFCEE is ≤20% for waters and ≤30% for soil samples.
LCL = Lower Control Limit
UCL = Upper Control Limit
LMEL = Lower Marginal Exceedance Limit
UMEL = Upper Marginal Exceedance Limit

Table 5
Characteristic Ions for Semi-volatile Compounds
Page 1 of 3

Compound	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
Bis(2-chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene-d_4 (I.S.)	152	150, 115
1,4-Dichlorobenzene	146	148, 111
Benzyl alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
N-Nitrosomethylethylamine	88	42, 88, 43, 56
Bis(2-chloroisopropyl)ether	45	77, 121
n-Nitroso-di-n-propylamine	70	42, 101, 130
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	95, 138
N-Nitrosodiethylamine	102	102, 42, 57, 44, 56
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Bis(2-chloroethoxy)methane	93	95, 123
Benzoic acid	122	105, 77
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene-d_8 (I.S.)	136	68
Naphthalene	128	129, 127
Hexachlorobutadiene	225	223, 227
4-Chloro-3-Methylphenol	107	144, 142
2-Methylnaphthalene	142	141
2-Methylphenol	107	107, 108, 77, 79, 90
Hexachlorocyclopentadiene	237	235, 272
N-Nitrosopyrrolidine	100	100, 1, 42, 68, 69
Acetophenone	105	71, 105, 51, 120
4-Methylphenol	107	107, 108, 77, 79, 90

Table 5
Characteristic IONS for Semivolatile Compounds
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Compound	Primary Ion	Secondary Ion(s)
2,4,6-Trichlorophenol	196	198, 200
2-Chloronaphthalene	162	127, 164
1-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethyl phthalate	163	194, 164
Acenaphthalene	152	151, 153
2,6-Dinitrotoluene	165	63, 89
3-Nitroaniline	138	108, 92
Acenaphthene-d_{10} (I.S.)	164	162, 160
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
2,6-Dinitrophenol	162	162, 164, 126, 98, 63
4-Chloroaniline	127	127, 129, 65, 92
Dibenzofuran	168	139
4-Nitrophenol	139	109, 65
Diethyl phthalate	149	177, 150
Fluorene	166	165, 167
N-Nitrosodibutylamine	84	84, 57, 41, 116, 158
4-Chlorophenyl phenyl ether	204	206, 141
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	168, 167
2,4,5-Trichlorophenol	196	196, 198, 97, 132, 99
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
4-Nitroaniline	138	138, 65, 108, 92, 80, 39
Phenanthrene-d_{10} (I.S.)	188	94, 80
Phenanthrene	178	179, 176
Anthracene	178	176, 179
1,4-Dinitrobenzene	168	168, 75, 50, 76, 92, 122
Di-n-butyl phthalate	149	150, 104
Fluoranthene	202	101, 203

Table 5
Characteristic IONS for Semivolatile Compounds
Page 3 of 3

Compound	Primary Ion	Secondary Ion(s)
Pyrene	202	200, 203
Butyl benzyl phthalate	149	91, 206
4-Nitrobiphenyl	199	199, 152, 141, 169, 151
Benz(a)anthracene	228	229, 226
Chrysene-d_{12} (I.S.)	240	120, 236
3,3'Dichlorobenzidine	252	254, 126
Chrysene	228	226, 229
Bis(2-ethylhexyl)phthalate	149	167, 279
3,3-Dimethylbenzene	212	212, 106, 196, 180
Di-n-octyl phthalate	149	167, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Perylene-d_{12} (I.S.)	264	260, 265
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenz(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
Pyridine	79	52
Aniline	93	62, 65
Biphenyl	154	153, 76
Carbazole	162	166, 139

Table 6

Summary of Method Quality Objectives for Method 8270C
Semi-Volatile Organic

QC Element	Frequency	Criteria	Corrective Action
GC/MS Tunes with DFTPP	Every 12 hours before analysis of standards and samples.	• Criteria listed in Table 2 • DDT breakdown < 20% • Peak tailing Pentachlorophenol <5 and Benzidine <3 • ***Tune in full scan mode for SIM analysis***	• Suspend all analysis until DFTPP non-compliance is rectified. • Report DDT breakdown and peak tailing exceedances in Project narrative.
Initial Calibration	Instrument set up. Each time the ICV or CCV can not meet criteria.	• Minimum of 5 point for average RF or linear regression or minimum of 6 points for quadratic regression and contains all analytes • Low standard ≤ MRL • ***Full scan:*** RSD≤15%, R≥0.995,R^2≥0.99 (Do not force through zero for LR) ***SIM analysis:*** RSD≤20%, R≥0.99 (Do not force through zero for LR) • CCCs must be ≤ 30% • If greater than 20% of compounds have %RSD>30%, then recalibration is necessary. • For SIM analysis the laboratory must monitor a minimum of two ions per analyte for all target analytes, surrogates and internal standards.	• No allowance. Perform maintenance and recalibrate. • If the average response or linear regression are not used for analyte quantitation (e.g., use of quadratic equation), this must be noted in the case narrative with a list of the affected analysis.
ICV	Immediately following initial calibration.	• %Rec = 70-130%. (75-125% DoD/Navy/AFCEE) • Must contain all target analytes. • Allow a maximum of 5 sporadic failures for full target list with expanded criteria of 50-150%. (No sporadic failures allowed for DoD/Navy/AFCEE/ USACE)	• If criteria are exceeded then remake and re-analyze ICV. If second consecutive ICV is acceptable criteria then calibration is accepted, otherwise recalibrate.

CCV	Every 12 hours prior to sample analysis.	• Concentration level near midpoint of curve • Must contain all target analytes. • Percent difference or percent drift must be ≤ 20% for CCC and ≤ 30% for all other compounds. NOTE: For MCP, re-calibrate if CCC>20% or if >10% of all other analytes are >30% (7 compounds). DoD/Navy/AFCEE/USACE require all target compounds to be ≤20%. (***For SIM analysis all compounds must be ≤ 30%.***	• Re-analyze CCV. If second consecutive CCV is in criteria then calibration is verified. If above fails, analyst may analyze two consecutive CCV at two concentrations. If above fails re-calibrate system and re-analyze any sample analyzed after invalid CCV. ***Exception: If CCV is exhibiting high bias (concentration is higher than upper limit) then any samples that are non-detect for that analyte may be reported.***
Method Blank	One per analytical batch of 20 or fewer samples.	• Matrix specific • Analytes < MRL (< ½ MRL for DoD/Navy/AFCEE/ USACE for all compounds) except for common lab contaminants (phthalates), which must be less than 5x the MRL	• Report exceedance in the project narrative. • Any samples that are non-detect for that analyte may be reported. • Samples with concentrations that are 20x higher than the method blank may be reported. • Samples reported with a contaminated blank must be "B" flagged. • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.
Blank spike/ Blank spike duplicate	One per analytical batch of 20 or fewer samples.	• Prepared using standard source different than used for initial calibration • Concentration level should be between low and mid-level standard • Must contain all analytes • Matrix specific • Percent recoveries between 40-140 (45-135% USACE) for the BN compounds and between 30-130 (35-140% USACE) for the acid compounds. ***See Table 4 for DoD/Navy/AFCEE*** • %RPD is ≤20% for waters and ≤30% for soils (≤30% for all DoD/Navy samples) • Laboratories must develop in-house limits that are within above criteria.	• Report exceedance in the project narrative. • Re-extract associated samples if >20% of all compounds are outside acceptance criteria (15 compounds) of if >15% from a particular class fall outside criteria (>3 acid compounds or >9 B/N compounds). • ***DoD/Navy/AFCEE/USACE: Allow a maximum of 4 sporadic failures for all targets. USACE expanded criteria is 15-150% for waters and 25-150% for soils. See Table 4 for DoD/Navy/ AFCEE expanded criteria.*** • Re-extract if the above exceptions do not apply. If re-extract is within hold, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client.

Matrix Spike	One per analytical batch of 20 or fewer samples	• Prepared using the same source as blank spikes • Concentration level should be between low and mid-level standard • Must contain all analytes • Matrix specific • Percent recoveries between 40-140 for the B/N compounds and between 30-130 for the acid compounds. Laboratories may develop in-house limits. ***USACE: Matrix spike % Recoveries are to be 45-135. See Table 4 for DoD/Navy/AFCEE*** • RPD should be < 20% for waters and < 30% for soils. (≤30% for all DoD/Navy samples)	• Check BS/BSD, if recoveries are acceptable then note exceedance in project narrative.
Surrogates	Added to all samples and standards.	• Use a minimum of 3 BN and 3 acid surrogates. • Percent recovery of 30-130% in soils. • Percent recovery in aqueous of 30-130% for B/N surrogates and 15-110% for acid surrogates. • ***USACE: 45-135% for B/N surrogates and 35-140% for Acid surrogates. See Table 4 for DoD/Navy/AFCEE*** • Laboratories must develop in-house limits that are within above criteria.	• ESS Laboratory policy allows for one BN and one acid surrogate to be outside criteria. If the recovery is <10% for any surrogate, then re-extraction is necessary. • If a surrogate is diluted to a concentration below the lowest standard, then no corrective action is needed. • If outside criteria (allowing for above exception) re-extract. • If surrogates are outside criteria for re-extract, report both sets of data. • If re-extract is within hold and within criteria, report just the re-extracted data. If re-extract is outside hold then report both sets of data to client. • Note exceedance in project narrative. If sample is not re-analyzed due to obvious interference (e.g., UCM), the chromatogram is to be included in the final report.

Internal Standards	Added to all samples and standards.	• Use a minimum of 6 across GC run. • Area counts in samples must be 50-200% of the area counts in associated CCV. • Retention times must be within ±30 sec of RT in associated CCV. See expanded criteria in Section 11.0.	• Re-analyze sample unless obvious interference is present (e.g., UCM) • Note exceedance in project narrative. • If IS is outside criteria for re-analysis, report both sets of data. • If re-analysis is within hold and within criteria, report just the re-analysis data. If re-analysis is outside hold then report both sets of data to client. • If re-analysis is not performed due to obvious contamination, then the laboratory must provide chromatogram.

16.0 DEFINITIONS

16.1 **Accuracy**: The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and of a common systematic error (or bias) component.

16.2 **Batch**: A group of samples which behave similarly with respect to the sampling or the testing procedures being employed and which are processed as a unit. For QC purposes, if the number of samples in a group is greater than 20, then each group of 20 samples or less will all be handled as a separate batch.

16.3 **Bias**: The deviation due to matrix effects of the measured value (x_s - x_u) from a known spiked amount, where x_s is the spiked sample and x_u is the un-spiked sample. Bias can be assessed by comparing a measured value to an accepted reference value in a sample of known concentration or by determining the recovery of a known amount of contaminant spiked into a sample (matrix spike).

16.4 **Control Sample**: A QC sample introduced into a process to monitor the performance of the system.

16.5 **Equipment Blank**: A sample of analyte-free media which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This blank is useful in documenting adequate decontamination of sampling equipment.

16.6 **Method Reporting Limit**: The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The MRL is generally 5 to 10 times the MDL. ESS Laboratory sets the MRL to the lowest non-zero standard in the calibration curve or higher.

16.7 **Field Duplicates**: Independent samples which are collected as close as possible to the same point in space and time. They are two separate samples taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the precision of the sampling process.

16.8 **Blank Spike (BS)**: A known matrix spiked with compound(s) representative of the target analytes. This is used to document laboratory performance.

16.9 **Matrix**: The component or substrate (e.g., surface water, drinking water) which contains the analyte of interest.

16.10 **Matrix Duplicate**: An intra-laboratory split sample which is used to document the precision of a method in a given sample matrix.

16.11 **Matrix Spike**: An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs prior to sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

16.12 **Matrix Spike Duplicates**: Intra-laboratory split samples spiked with identical concentrations of target analyte(s). The spiking occurs prior to sample preparation and analysis. They are used to document the precision and bias of a method in a given sample matrix.

16.13 **Method Blank**: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

16.14 **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 and is determined from analysis of a sample in a given matrix type containing the analyte. See SOP 110_0013 for further explanation.

16.15 **Organic-Free Reagent Water**: For volatiles, all references to water in the method refer to water in which an interferant is not observed at the method detection limit of the compounds of interest. A water purification system is used to generate organic-free deionized water.

16.16 **Records**: Include all logbooks, papers, machine readable materials, or other documentary materials, regardless of physical form or characteristics.

16.17 **Surrogate**: An organic compound which is similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

16.18 **Trip Blank**: A sample of analyte-free media taken from the laboratory to the sampling site and returned to the laboratory unopened. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of blank is useful in documenting contamination of volatile organics samples.

17.0 PERSONNEL QUALIFICATIONS

17.1 Analysts who perform this analysis must have a working knowledge or quantitative and qualitative analysis, instrumental methods of analysis, chemical laboratory methods, and equipment.

17.2 All analysts, before performing any analysis, participate in the ESS Laboratory training program (SOP80_0016). The training process consists of reading the Standard Operating Procedure, gaining instruction on the procedure from an experienced analyst, and performing the initial demonstration of capability.

18.0 TROUBLESHOOTING

18.1 **Daily Maintenance**: The following maintenance is performed prior to calibration or sample analysis. The purpose of this maintenance is to clean the injection system.

- Cool injection port and oven to room temperature.
- Turn off gas flow by setting EPC to 0.0. Access EPC by pressing the yellow shift key on the GC control panel then hitting the Injector B Temp button.
- Disconnect the column and cap the end with old septa.
- Remove septum nut and weldment.
- Take out liner and clean with methylene chloride using a cotton swab.
- Clean gold seal and inside the injection port.
- Re-attach the gold seal, replace liner and o-ring.
- Clean weldment Inlet and septum holder.
- Clean septum nut.
- Screw on weldment and replace septa if needed. The septa nut should be finger tight.
- Clip off about 6" of column and re-connect.
- Set EPC to 7.0, bake out injection port at 275°C and set oven temperature to 320°C for 30 minutes.
- Clean Injection syringe and guide
- Record daily maintenance in the Semi-Volatile maintenance logbook, Attachment D.

18.2 If DFTPP criteria are not met, the analysis must be repeated. Sample analysis can not begin until DFTPP meets acceptance criteria. Repeated failures indicate that the MS acquisition parameters must be adjusted. These parameters should be adjusted in manual tune, saved and the tune repeated.

18.3 If manual or auto-tune does not produce DFTPP spectra within acceptance criteria, the MS source may need cleaning. Cleaning instructions are found in the MS detector manual.

18.4 See laboratory supervisor or operations manager for all other maintenance problems.

18.5 Record all maintenance in the instrument's maintenance logbook.

19.0 DATA MANAGEMENT AND RECORDS

19.1 **Data Management** - ESS Laboratory utilizes the Promium Element LIMS system as part of its Data Management system. Client sample information is entered into

ELEMENT LIMS and analyses are assigned to each sample. The LIMS allows EPA hold times, minimum batch QC requirements, and QC criteria to be assigned to each analysis. Standards can be entered and assigned to QC samples through the LIMS. Once analysis has been performed, data is imported using DataTool avoiding manual errors. In conjunction with Crystal Reports, the ELEMENT system allows for a wide variety of reporting formats.

19.2 **Records** – The specific retention periods required in the NELAC Standards, EPA-CFR and state and local statutes are followed or exceeded. At a minimum, data records are retained for five years from last use (10 years for drinking water). If there is a question about whether a record should be retained or disposed because no specific requirement could be found, the record is retained until such time as a retention period is specified. Records are stored in specified-labeled locations and are easily retrievable. All raw data associated with testing is also retained including; computer printouts, chromatograms, review forms, and logbooks.

20.0 ATTACHMENTS

Appendix A Low Level PAH and Pentachlorophenol analysis

Appendix B Certificates of Analysis

Appendix C Enviroquant Method

Appendix D MS 1 & MS2 Sequence Logs and Maintenance Record

Appendix E Lab Data Review Checklist

Appendix F Exception Sheet

Appendix A

SemiVolatile Organic Compounds Method 8270

Low Level PAH and Pentachlorophenol Analysis

SOP 60_8270C Addendum

Method Summary: Aqueous samples are extracted by EPA Method 3510C. Extracts are concentrated to a final volume of 0.25 mls. Soil samples are extracted by EPA Methods 3541 or 3546 and concentrated to a final volume of 0.5 ml. Samples are analyzed by GC/MS in selective ion monitoring mode to achieve low detection limits. (0.05 µg/L for aqueous samples and 16.7 µg/Kg for soil samples).

The following are the 8270 modifications. Refer to SOP 60_ 8270 and appropriate preparation method SOP for all other procedures.:

I. Standards

a. Stock: A 20ppm intermediate stock is prepared from the 200ppm 8270 stock by diluting 1ml to 10 ml with methylene chloride in a 10ml volumetric flask. This applies to the primary and second source standards.

b. Working Standards:

Standard	Amount of Stock Added (ul)	Final Volume ml	Final Conc. On Column ug/ml
Level 0.2	10	1	0.2
Level 0.4	20	1	0.4
Level 0.8	40	1	0.8
Level 1.0	50	1	1.0
Level 2.0	100	1	2.0
Level 4.0	200	1	4.0
Level 8.0	400	1	8.0
Level 10	500	1	10
Level 20	1000	1	20
SS PAH 1.0	50	1	1.0
SS PCP 5.0	250	1	5.0
CCV PAH 1.0	50	1	1.0
CCV PCP 5.0	250	1	5.0

c. Internal Standard: The 4000ppm 8270 standard is diluted 10x to 400ppm. (100µl to 1ml in methylene chloride).

d. Surrogate Standard: A surrogate spiking solution is prepared at 2.5 µg/ml. Prepared by diluting 2.5 ml of the 8270 surrogate to 100 ml with acetone in a volumetric flask.
   i. Aqueous samples and QC spike volume is 250µl.
   ii. Soil Samples and QC spike is 500µl.
e. PAH matrix spike solution: Prepared at 2.5 µg/ml. Prepared by diluting 2.5 ml of the 8270 Matrix Spike to 100 ml with acetone in a volumetric flask
   i. Aqueous samples andQC spike 250µl.
   ii. Soil samples and QC spike 500µl.
f. Pentachlorophenol Spike: (Aqueous samples only) Use the SVOA matrix spike at 100µg/ml. (The high concentration standard maintains the integrity of the PCP) For aqueous samples spike 25µl into the BS/BSD/MS/MSD. By concentrating the extract to 0.25 ml, this will produce an on column concentration of 10µg/ml.

II. Extraction:
a. Aqueous: Up to 1L of sample is shaken by separatory funnel (3510C) with methylene chloride using a base -> acid extraction. The appropriate surrogates and spikes are added. The extract is concentrated to a final volume of 0.25ml in an NEVAP and measured with a 500 ml graduated Hamilton syringe. The extract is transferred to a microinsert in a GC target vial for analysis. The vial is capped. 2.5 µl of the 400ppm internal standard is spiked into the 250µl extract. (10µl is added to each 1ml standard.) This will produce a 4ppm on column concentration of Internal Standard.
b. Soil: 15grams is extracted by EPA Method 3541 or 3546. The appropriate surrogates and spikes are added. The extract is concentrated to 0.5ml and transferred to a GC target vial and capped. 5µl of the 400 ppm internal standard is spiked into each extract.

III. Analysis:
a. Tune: Performed in full scan mode. Ion ratios evaluated and must meet 8270 criteria. Analyzed prior to analysis, every 12 hours.
b. ICAL: All analytes <20% or Corr >= 0.99. Quad only acceptable for PCP and must be > 0.99 corr.
c. SCV: 70-130% recovery. No failures.
d. CCV: 70-130% No failures. Analyzed prior to sample analysis with no failures.
e. All analytes including surrogates and internal standards analyzed in selective ion monitoring mode. The following is a list of analytes and ions scanned:

PAH SIMS

Group	Analytes	Type	Ions	Abs RT	Start Scan Time	Stop Scan Time
1	Baseline		74	1.00	1.00	
2	1,4-DCB-d4	IS	152,115,150	3.38	2.88	
	1,2-DCB-d4	Surr		3.56		
3	Nitrobenzene-d5	Surr	82,128,54	3.94	3.75	4.32
4	Napthalene-d8	IS	136,68,128,129	4.70	4.32	5.17
	Napthalene	Trgt		4.72		
5	2-Methylnaphthalene	Trgt	172,171,142,141	5.61	5.17	6.61
	1-Methylnaphthalene	Trgt		5.77		
	2-Fluorobiphenyl	Surr		6.19		
6	Acenaphthylene	Trgt	152,153,154,164	7.02	6.61	7.81
	Acenaphthene-d10	IS		7.29		
	Acenaphthene	Trgt		7.35		
7	Fluorene	Trgt	165,166,167	8.26	7.81	8.61
8	2,4,6-Tribromophenol	Surr	330,332	8.95	8.61	9.23
	Pentachlorophenol	Trgt		9.50		
9	Phenanthrene-d10	IS	176,178,188	10.08		
	Phenanthrene	Trgt	266,264	10.12		
	Anthracene	Trgt		10.21	9.23	11.40
10	Fluoranthene	Trgt	202,100,101	12.58	11.40	13.25
	Pyrene	Trgt		13.04		
11	Terphenyl-d14	Surr	122,212,244	13.46	13.25	14.53
12	Benzo(a)anthracene	Trgt	226,228,229,240	15.59	14.53	16.72
	Chrysene-d12	IS		15.62		
	Chrysene	Trgt		15.68		
13	Benzo(b)	Trgt	252,253,125,264	17.75	16.72	19.37
	Benzo(k)	Trgt		17.80		
	Benzo(a)pyrene	Trgt		18.34		
	Perylene-d12	IS		18.44		
14	Indeno 123	Trgt	276,278,138,139	20.29	19.37	to END
	Dibenzo(ah)	Trgt		20.31		
	Benzo(ghi)	Trgt		20.75		

IV: Reporting: On column results in µg/ml. Final results in ppb are calculated using initial and final volumes. See SOP 60_8270.

LBIS0136 Appendix B.

Certificate of Analysis

DESCRIPTION: EPA TCLP Acids Mix

CATALOG NO.: 48142 MFG DATE: Apr-2005

LOT NO.: LB29273 EXPIRATION DATE: Apr-2008

SOLVENT: METHANOL

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
PENTACHLOROPHENOL	87-86-5	99.9	999	1030	+/-	20.2	LB01443
2-METHYLPHENOL	95-48-7	99.2	1001	1010	+/-	3.6	LA39009
2,4,5-TRICHLOROPHENOL	95-95-4	99.9	1001	993	+/-	17.7	LA40299
2,4,6-TRICHLOROPHENOL	88-06-2	99.2	999	992	+/-	15.3	LB09687
3-METHYLPHENOL	108-39-4	99.8	1000	974	+/-	3.7	LB08578
4-METHYLPHENOL	106-44-5	99.8	1002	1021	+/-	1.2	LA39011

(1) Listed in alphabetical order.

(2) Determined by capillary GC-FID, unless otherwise noted.

(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty

wood Doughty
uality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

6B15135

Certificate of Analysis

DESCRIPTION: EPA 1311 Base-Neutrals

CATALOG NO.: 48947 MFG DATE: May-2005

LOT NO.: LB29695 EXPIRATION DATE: May-2008

SOLVENT: ACETONE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
HEXACHLOROBENZENE	118-74-1	99.9	1002	1012	+/-	2.6	LB09543
HEXACHLOROBUTADIENE	87-68-3	98.5	1004	1011	+/-	3.9	LA95300
HEXACHLOROETHANE	67-72-1	99.9	1002	996	+/-	7.2	LB29072
NITROBENZENE	98-95-3	99.9	1004	1010	+/-	4.6	LA39293
2,4-DINITROTOLUENE	121-14-2	99.3	1004	1006	+/-	4.4	LA39290

(1) Listed in alphabetical order.

(2) Determined by capillary GC-FID, unless otherwise noted.

(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

GB15092-96

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31083 **Lot No.:** A039542

Description: Acid Surrogate Standard Mix (3/90)

Expiration Date[1]: March 2010 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Fluorophenol	367-12-4	99%	7500 ug/mL	+/- 0.1
2	Phenol-d6	13127-88-3	97%	7500 ug/mL	+/- 0.1
3	2-Chlorophenol-d4	93951-73-6	99%	7500 ug/mL	+/- 0.1
4	2,4,6-Tribromophenol	118-79-6	99%	7500 ug/mL	+/- 0.1
	Solvent: Methanol	67-56-1	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

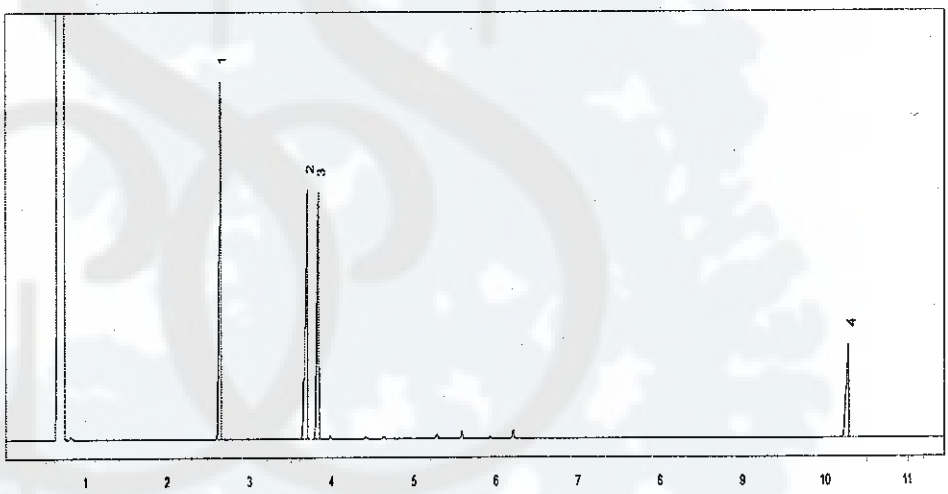
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
50°C to 300°C
@ 15°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: GD

John Udgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

6B15087-91

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31082 **Lot No.:** A039546

Description: B/N Surrogate Standard Mix (3/90)

Expiration Date[1]: October 2008 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,2-Dichlorobenzene-d4	2199-69-1	99%	5000 ug/mL	+/- 0.04
2	Nitrobenzene-d5	4165-60-0	99%	5000 ug/mL	+/- 0.04
3	2-Fluorobiphenyl	321-60-8	99%	5000 ug/mL	+/- 0.04
4	p-Terphenyl-d14	1718-51-0	99%	5000 ug/mL	+/- 0.04
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

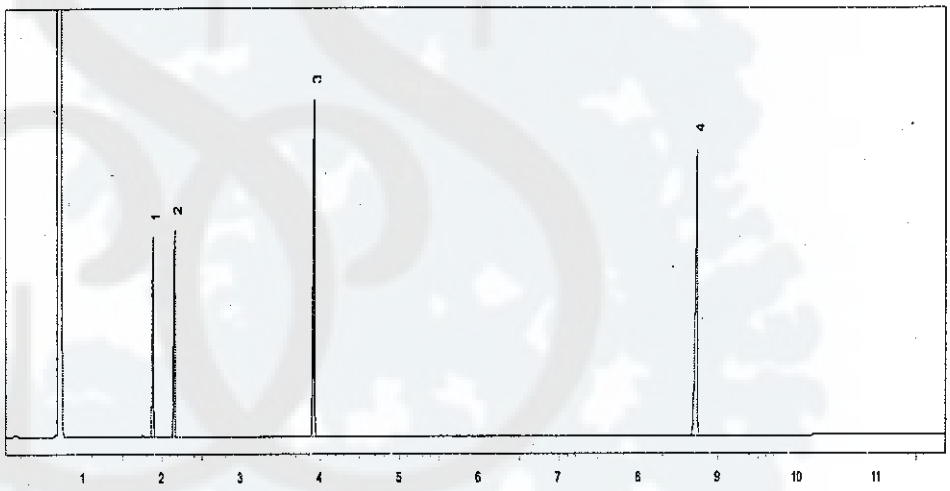
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
100°C to 330°C
@ 20°C/min. (hold 10 min.)

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: GD

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

6B13020-24

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31006 **Lot No.:** A039209

Description: SV Internal Standard Mix

Expiration Date[1]: September 2012 **Storage:** Refrigerate

Handling: Warm & Sonicate prior to use

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Dichlorobenzene-d4	3855-82-1	99%	4000 ug/mL	+/- 0.04
2	Naphthalene-d8	1146-65-2	97%	4000 ug/mL	+/- 0.04
3	Acenaphthene-d10	15067-26-2	99%	4000 ug/mL	+/- 0.04
4	Phenanthrene-d10	1517-22-2	97%	4000 ug/mL	+/- 0.04
5	Chrysene-d12	1719-03-5	98%	4000 ug/mL	+/- 0.04
6	Perylene-d12	1520-96-3	99%	4000 ug/mL	+/- 0.04
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.# 10238)

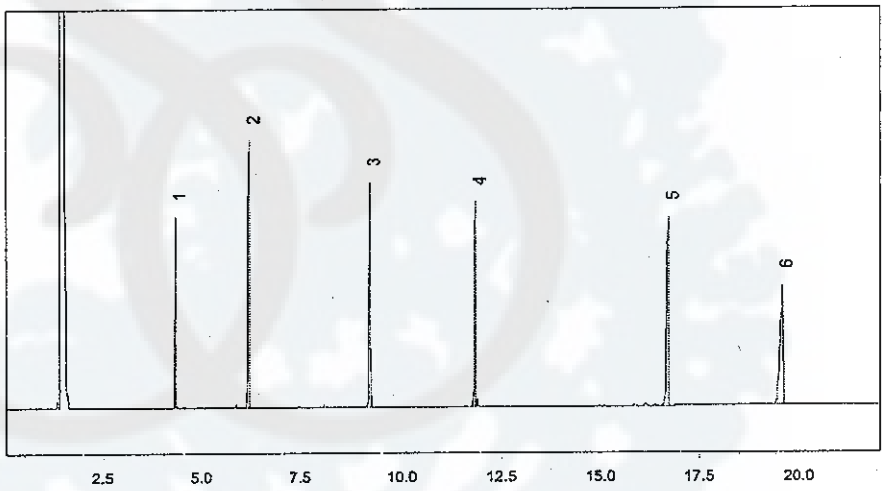
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
75°C to 330°C
@ 15°C/min. (hold 5 min.)

Inj. Temp:
250°C

Det. Temp:
330°C

Det. Type:
FID



Manufactured By: GD

John Lidgett

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

8270 Primary Mix1 (1 of 4) 6A31070 6A31071

RESTEK

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31850 **Lot No.:** A042069

Description: 8270 MegaMix

Expiration Date[1]: June 2007 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Pyridine	110-86-1	99%	1000 ug/mL	+/- 0.2
2	N-Nitrosodimethylamine	62-75-9	99%	1000 ug/mL	+/- 0.2
3	Aniline	62-53-3	99%	1000 ug/mL	+/- 0.2
4	Phenol	108-95-2	99%	1000 ug/mL	+/- 0.2
5	Bis(2-chloroethyl)ether	111-44-4	99%	1000 ug/mL	+/- 0.2
6	2-Chlorophenol	95-57-8	99%	1000 ug/mL	+/- 0.2
7	1,3-Dichlorobenzene	541-73-1	99%	1000 ug/mL	+/- 0.2
8	1,4-Dichlorobenzene	106-46-7	99%	1000 ug/mL	+/- 0.2
9	1,2-Dichlorobenzene	95-50-1	99%	1000 ug/mL	+/- 0.2
10	Benzyl alcohol	100-51-6	99%	1000 ug/mL	+/- 0.2
11	Bis(2-chloroisopropyl)ether	108-60-1	99%	1000 ug/mL	+/- 0.2
12	2-Methylphenol (o-cresol)	95-48-7	99%	1000 ug/mL	+/- 0.2
13	Hexachloroethane	67-72-1	99%	1000 ug/mL	+/- 0.2
14	N-Nitroso-di-n-propylamine	621-64-7	99%	1000 ug/mL	+/- 0.2
15	4-Methylphenol (p-cresol)	106-44-5	99%	500 ug/mL	+/- 0.2
16	3-Methylphenol (m-cresol)	108-39-4	99%	500 ug/mL	+/- 0.2
17	Nitrobenzene	98-95-3	99%	1000 ug/mL	+/- 0.2
18	Isophorone	78-59-1	99%	1000 ug/mL	+/- 0.2
19	2-Nitrophenol	88-75-5	99%	1000 ug/mL	+/- 0.2
20	2,4-Dimethylphenol	105-67-9	99%	1000 ug/mL	+/- 0.2
21	Bis(2-chloroethoxy)methane	111-91-1	99%	1000 ug/mL	+/- 0.2
22	2,4-Dichlorophenol	120-83-2	99%	1000 ug/mL	+/- 0.2
23	1,2,4-Trichlorobenzene	120-82-1	99%	1000 ug/mL	+/- 0.2
24	Naphthalene	91-20-3	99%	1000 ug/mL	+/- 0.2
25	4-Chloroaniline	106-47-8	99%	1000 ug/mL	+/- 0.2
26	Hexachloro-1,3-butadiene (Hexachlorobutadiene)	87-68-3	99%	1000 ug/mL	+/- 0.2
27	4-Chloro-3-methylphenol	59-50-7	99%	1000 ug/mL	+/- 0.2
28	2-Methylnaphthalene	91-57-6	98%	1000 ug/mL	+/- 0.2
29	1-Methylnaphthalene	90-12-0	99%	1000 ug/mL	+/- 0.2
30	Hexachlorocyclopentadiene	77-47-4	99%	1000 ug/mL	+/- 0.2
31	2,4,6-Trichlorophenol	88-06-2	99%	1000 ug/mL	+/- 0.2
32	2,4,5-Trichlorophenol	95-95-4	98%	1000 ug/mL	+/- 0.2
33	2-Chloronaphthalene	91-58-7	99%	1000 ug/mL	+/- 0.2
34	2-Nitroaniline	88-74-4	99%	1000 ug/mL	+/- 0.2
35	1,4-Dinitrobenzene	100-25-4	99%	1000 ug/mL	+/- 0.2
36	Acenaphthylene	208-96-8	99%	1000 ug/mL	+/- 0.2
37	1,3-Dinitrobenzene	99-65-0	99%	1000 ug/mL	+/- 0.2
38	Dimethylphthalate	131-11-3	99%	1000 ug/mL	+/- 0.2
39	2,6-Dinitrotoluene	606-20-2	99%	1000 ug/mL	+/- 0.2
40	1,2-Dinitrobenzene	528-29-0	99%	1000 ug/mL	+/- 0.2
41	Acenaphthene	83-32-9	99%	1000 ug/mL	+/- 0.2
42	3-Nitroaniline	99-09-2	99%	1000 ug/mL	+/- 0.2
43	2,4-Dinitrophenol	51-28-5	98%	1000 ug/mL	+/- 0.2
44	Dibenzofuran	132-64-9	99%	1000 ug/mL	+/- 0.2
45	2,4-Dinitrotoluene	121-14-2	98%	1000 ug/mL	+/- 0.2
46	4-Nitrophenol	100-02-7	99%	1000 ug/mL	+/- 0.2
47	2,3,4,6-Tetrachlorophenol	58-90-2	99%	1000 ug/mL	+/- 0.2
48	2,3,5,6-Tetrachlorophenol	935-95-5	99%	1000 ug/mL	+/- 0.2
49	Fluorene	86-73-7	99%	1000 ug/mL	+/- 0.2
50	4-Chlorophenyl phenyl ether	7005-72-3	99%	1000 ug/mL	+/- 0.2
51	Diethylphthalate	84-66-2	99%	1000 ug/mL	+/- 0.2
52	4-Nitroaniline	100-01-6	99%	1000 ug/mL	+/- 0.2

53	4,6-Dinitro-2-methylphenol (Dinitro-o-cresol)	534-52-1	99%	1000 ug/mL	+/- 0.2
54	Diphenylamine	122-39-4	99%	1000 ug/mL	+/- 0.2
55	Azobenzene	103-33-3	98%	1000 ug/mL	+/- 0.2
56	4-Bromophenyl phenyl ether	101-55-3	99%	1000 ug/mL	+/- 0.2
57	Hexachlorobenzene	118-74-1	99%	1000 ug/mL	+/- 0.2
58	Pentachlorophenol	87-86-5	97%	1000 ug/mL	+/- 0.2
59	Phenanthrene	85-01-8	99%	1000 ug/mL	+/- 0.2
60	Anthracene	120-12-7	99%	1000 ug/mL	+/- 0.2
61	Carbazole	86-74-8	98%	1000 ug/mL	+/- 0.2
62	Di-n-butylphthalate	84-74-2	99%	1000 ug/mL	+/- 0.2
63	Fluoranthene	206-44-0	98%	1000 ug/mL	+/- 0.2
64	Pyrene	129-00-0	99%	1000 ug/mL	+/- 0.2
65	Benzyl butyl phthalate	85-68-7	99%	1000 ug/mL	+/- 0.2
66	Bis(2-ethylhexyl)adipate	103-23-1	99%	1000 ug/mL	+/- 0.2
67	Benz(a)anthracene	56-55-3	98%	1000 ug/mL	+/- 0.2
68	Chrysene	218-01-9	99%	1000 ug/mL	+/- 0.2
69	Bis(2-ethylhexyl)phthalate	117-81-7	99%	1000 ug/mL	+/- 0.2
70	Di-n-octyl phthalate	117-84-0	99%	1000 ug/mL	+/- 0.2
71	Benzo(b)fluoranthene	205-99-2	99%	1000 ug/mL	+/- 0.2
72	Benzo(k)fluoranthene	207-08-9	99%	1000 ug/mL	+/- 0.2
73	Benzo(a)pyrene	50-32-8	99%	1000 ug/mL	+/- 0.2
74	Indeno(1,2,3-cd)pyrene	193-39-5	99%	1000 ug/mL	+/- 0.2
75	Dibenz(a,h)anthracene	53-70-3	99%	1000 ug/mL	+/- 0.2
76	Benzo(g,h,i)perylene	191-24-2	99%	1000 ug/mL	+/- 0.2
	Solvent: Methylene Chloride (MEOH FREE)	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

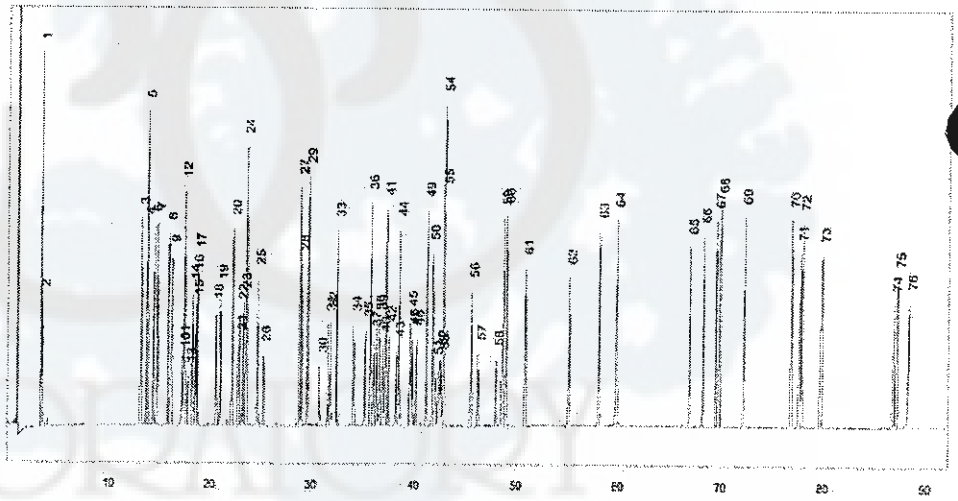
Carrier Gas:
hydrogen @ 40cm/sec.

Temp. Program:
35°C (hold 3 min.) to 330°C
@ 3°C/min

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: FJT

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED
Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

Tech Tip:

N-Nitrosodiphenylamine is prone to breakdown in the injection port and will be converted to diphenylamine. N-Nitrosodiphenylamine is also a reactive species that can initiate premature decomposition of other compounds in the mix. For these reasons diphenylamine is used in the preparation of this mixture. When comparing the response of this compound to mixtures manufactured using N-nitrosodiphenylamine, a difference in response will be observed.

8270 Primary Mix 2 (2 of 4)

6A31087
6A31088

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31806 **Lot No.:** A042422

Description: Appendix IX Mix #2

Expiration Date[1]: July 2007 **Storage:** Freezer

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	1,4-Dioxane	123-91-1	99%	1000 ug/mL	+/- 0.1
2	Ethyl methacrylate	97-63-2	99%	1000 ug/mL	+/- 0.1
3	Methyl methanesulfonate	66-27-3	99%	1000 ug/mL	+/- 0.1
4	Ethyl methanesulfonate	62-50-0	99%	1000 ug/mL	+/- 0.1
5	Benzaldehyde	100-52-7	99%	1000 ug/mL	+/- 0.1
6	Pentachloroethane	76-01-7	98%	1000 ug/mL	+/- 0.1
7	Acetophenone	98-86-2	99%	1000 ug/mL	+/- 0.1
8	2,6-Dichlorophenol	87-65-0	99%	1000 ug/mL	+/- 0.1
9	Hexachloropropene	1888-71-7	99%	1000 ug/mL	+/- 0.1
10	epsilon-Caprolactam	105-60-2	99%	1000 ug/mL	+/- 0.1
11	Isosafrole (cis & trans)	120-58-1	99%	1000 ug/mL	+/- 0.1
12	1,2,4,5-Tetrachlorobenzene	95-94-3	99%	1000 ug/mL	+/- 0.1
13	Safrole	94-59-7	99%	1000 ug/mL	+/- 0.1
14	Biphenyl	92-52-4	99%	1000 ug/mL	+/- 0.1
15	1-Chloronaphthalene	90-13-1	86%	1000 ug/mL	+/- 0.1
16	Diphenyl ether	101-84-8	99%	1000 ug/mL	+/- 0.1
17	1,4-Naphthoquinone	130-15-4	99%	1000 ug/mL	+/- 0.1
18	Pentachlorobenzene	608-93-5	99%	1000 ug/mL	+/- 0.1
19	Diallate (cis and trans)	2303-16-4	99%	1000 ug/mL	+/- 0.1
20	1,3,5-Trinitrobenzene	99-35-4	98%	1000 ug/mL	+/- 0.1
21	Phenacetin	62-44-2	99%	1000 ug/mL	+/- 0.1
22	Atrazine	1912-24-9	99%	1000 ug/mL	+/- 0.1
23	Pentachloronitrobenzene (quintozene)	82-68-8	98%	1000 ug/mL	+/- 0.1
24	Pronamide (Propyzamide)	23950-58-5	99%	1000 ug/mL	+/- 0.1
25	4-Nitroquinoline-N-oxide	56-57-5	98%	1000 ug/mL	+/- 0.1
26	Isodrin	465-73-6	99%	1000 ug/mL	+/- 0.1
27	Aramite	140-57-8	--------%	1000 ug/mL	+/- 0.1
28	Chlorobenzilate	510-15-6	99%	1000 ug/mL	+/- 0.1
29	Kepone	143-50-0	99%	1000 ug/mL	+/- 0.1
30	7,12-Dimethylbenz(a)anthracene	57-97-6	99%	1000 ug/mL	+/- 0.1
31	3-Methylcholanthrene	56-49-5	99%	1000 ug/mL	+/- 0.1
32	Dibenz(a,j)acridine	224-42-0	99%	1000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride (MEOH FREE)	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

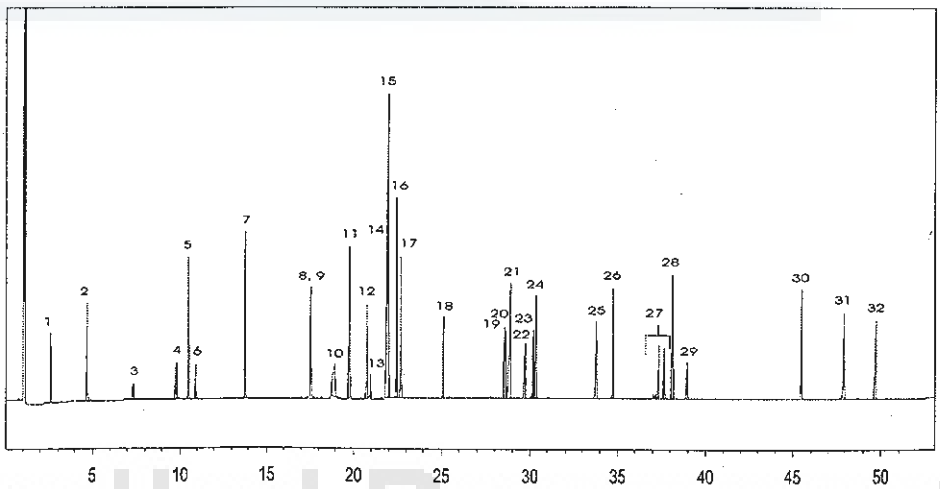
Carrier Gas:
helium @ 1 ml/min.

Temp. Program:
35°C (hold 4 min.) to 330°C
@ 6°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



8270 Primary Mix 3 (3 of 4) Restek 6A31089

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31852 **Lot No.:** A039352

Description: 8270 Benzidines Mix #2

Expiration Date[1]: October 2008 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Benzidine	92-87-5	99%	2000 ug/mL	+/- 0.1
2	3,3'-Dimethylbenzidine (o-tolidine)	119-93-7	99%	2000 ug/mL	+/- 0.1
3	3,3'-Dichlorobenzidine	91-94-1	99%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride (MEOH FREE)	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.#10238)

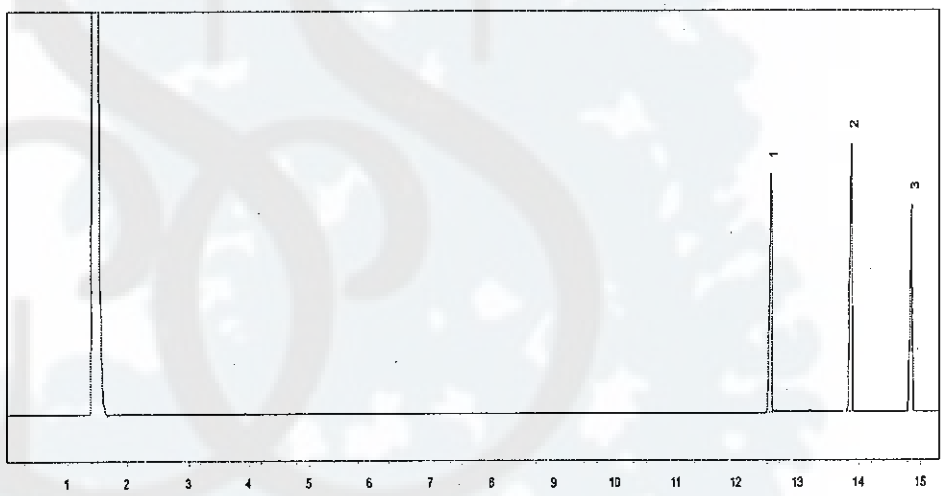
Carrier Gas:
hydrogen @ 40 cm./sec.

Temp. Program:
100°C to 330°C
@ 15°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: GD

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

CA31091-12

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31879 **Lot No.:** A036666

Description: Benzoic Acid Mix

Expiration Date[1]: April 2009 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	Benzoic acid	65-85-0	99%	2000 ug/mL	+/- 0.1
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.# 10238)

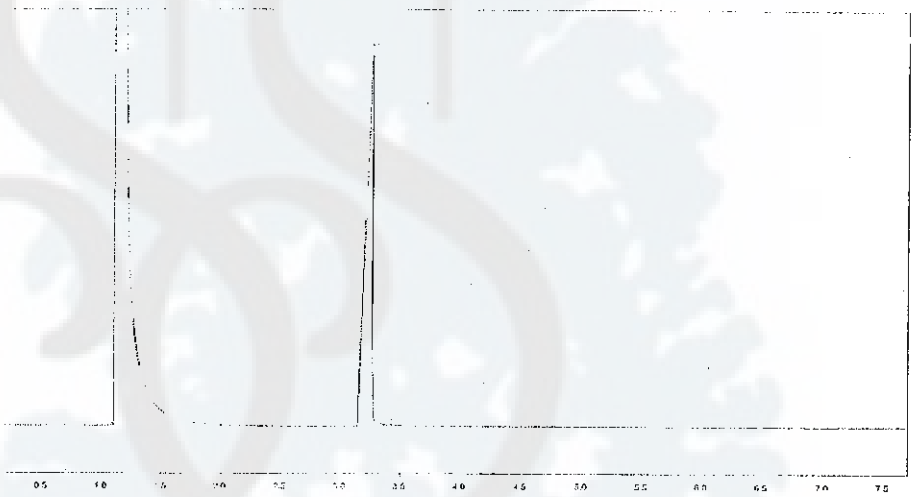
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
110°C to 225°C
@ 15°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: MWB

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System Certificate #FM80397

8270 55 Mix 1 (1 of 5) SVM-8270 CA 30065-68

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product SVM-8270
Lot Number: CB-2326

Expiration Date: Oct-2006
Page: 1 of 3

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
acenaphthene	000083-32-9	29697-41	1002 ± 5 µg/mL
acenaphthylene	000208-96-8	34267-35/ER11 1504-01	1002 ± 5 µg/mL
anthracene	000120-12-7	33383-91	1002 ± 5 µg/mL
benz[a]anthracene	000056-55-3	34764-06	1002 ± 5 µg/mL
benzo[b]fluoranthene	000205-99-2	33385-53	1002 ± 5 µg/mL
benzo[k]fluoranthene	000207-08-9	34750-27	1002 ± 5 µg/mL
benzo[ghi]perylene	000191-24-2	34750-40	1002 ± 5 µg/mL
benzo[a]pyrene	000050-32-8	02903HB	1002 ± 5 µg/mL
carbazole	000086-74-8	F26K06	1002 ± 5 µg/mL
chrysene	000218-01-9	33385-57	1002 ± 5 µg/mL
dibenz[a,h]anthracene	000053-70-3	35091-73	1002 ± 5 µg/mL
fluoranthene	000206-44-0	34260-17	1002 ± 5 µg/mL
fluorene	000086-73-7	29240-49	1002 ± 5 µg/mL
indeno[1,2,3-cd]pyrene	000193-39-5	ER102604-03	1002 ± 5 µg/mL
naphthalene	000091-20-3	14205KB	1002 ± 5 µg/mL
phenanthrene	000085-01-8	35034-93	1002 ± 5 µg/mL
pyrene	000129-00-0	22599-73	1002 ± 5 µg/mL
azobenzene	000103-33-3	DF-04606DF	1002 ± 5 µg/mL
4-chloroaniline	000106-47-8	PW-01310MW	1002 ± 5 µg/mL
2-chloronaphthalene	000091-58-7	FIE01	1002 ± 5 µg/mL
4-chloro-3-methylphenol	000059-50-7	NB-061657	1002 ± 5 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product SVM-8270 **Expiration Date:** Oct-2006
Lot Number: CB-2326 **Page:** 2 of 3

Analyte	CAS#	Analyte Lot	True Value
dibenzofuran	000132-64-9	KB-080477	1002 ± 5 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	1002 ± 5 µg/mL
2,4-dichlorophenol	000120-83-2	HZ-05729EZ	1002 ± 5 µg/mL
2-methyl-4,6-dinitrophenol	000534-52-1	4213614	1002 ± 5 µg/mL
2,4-dinitrophenol	000051-28-5	NT01854	1002 ± 5 µg/mL
2,4-dinitrotoluene	000121-14-2	18219TA	1002 ± 5 µg/mL
2,6-dinitrotoluene	000606-20-2	DU-08328CR	1002 ± 5 µg/mL
hexachlorobenzene	000118-74-1	EB-1480	1002 ± 5 µg/mL
hexachloroethane	000067-72-1	DN-06203HF	1002 ± 5 µg/mL
pentachlorophenol	000087-86-5	07119HO	1002 ± 5 µg/mL
2-nitrophenol	000088-75-5	N980025	1002 ± 5 µg/mL
4-nitrophenol	000100-02-7	HE-5518HE	1002 ± 5 µg/mL
2-nitroaniline	000088-74-4	AO-13201TU	1002 ± 5 µg/mL
3-nitroaniline	000099-09-2	HM-03020DL	1002 ± 5 µg/mL
4-nitroaniline	000100-01-6	KL-3926DK	1002 ± 5 µg/mL
2,4,5-trichlorophenol	000095-95-4	DO-07521CO	1002 ± 5 µg/mL
2,4,6-trichlorophenol	000088-06-2	35090-08	1002 ± 5 µg/mL
bis(2-chloroethyl) ether	000111-44-4	BC-13187	1002 ± 5 µg/mL
bis(2-chloroethoxy)methane	000111-91-1	330-26A	1002 ± 5 µg/mL
bis(2-ethylhexyl) phthalate	000117-81-7	D-23230	1002 ± 5 µg/mL
4-bromophenyl phenyl ether	000101-55-3	KO-05819CO	1002 ± 5 µg/mL
butyl benzyl phthalate	000085-68-7	EB-1716	1002 ± 5 µg/mL
4-chlorophenyl phenyl ether	007005-72-3	00617EU	1002 ± 5 µg/mL
2-chlorophenol	000095-57-8	CS-09106BQ	1002 ± 5 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product SVM-8270 **Expiration Date:** Oct-2006
Lot Number: CB-2326 **Page:** 3 of 3

Analyte	CAS#	Analyte Lot	True Value
di-n-butyl phthalate	000084-74-2	AC-011687	1002 ± 5 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	1002 ± 5 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	1002 ± 5 µg/mL
diethyl phthalate	000084-66-2	KU-04114MS	1002 ± 5 µg/mL
2,4-dimethylphenol	000105-67-9	BG-14822JF	1002 ± 5 µg/mL
dimethyl phthalate	000131-11-3	D-44220	1002 ± 5 µg/mL
di-n-octyl phthalate	000117-84-0	224-99A	1002 ± 5 µg/mL
hexachlorobutadiene	000087-68-3	339923/1	1002 ± 5 µg/mL
hexachlorocyclopentadiene	000077-47-4	KO-03907AU	1002 ± 5 µg/mL
isophorone	000078-59-1	PQ-10830MQ	1002 ± 5 µg/mL
2-methylnaphthalene	000091-57-6	15416DA	1002 ± 5 µg/mL
nitrobenzene	000098-95-3	HK-2925LK	1002 ± 5 µg/mL
N-nitrosodimethylamine	000062-75-9	PS-1207JS	1002 ± 5 µg/mL
N-nitrosodi-n-propylamine	000621-64-7	GA01	1002 ± 5 µg/mL
1,2,4-trichlorobenzene	000120-82-1	00334TQ	1002 ± 5 µg/mL
o-cresol	000095-48-7	JG-05301TZ	1002 ± 5 µg/mL
p-cresol	000106-44-5	KG-18923HG	1002 ± 5 µg/mL
bis(2-chloroisopropyl) ether	000108-60-1	PR-16041	1002 ± 5 µg/mL
phenol	000108-95-2	MY-05714AY	1002 ± 5 µg/mL

Matrix: methylene chloride/benzene (3:1)

2004 ± 5 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

8270 SS Mix2 (2 of 5) SVM-8271

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis 6A31039-42

Semi-Volatiles Mixture

Product SVM-8271
Lot Number: CB-2681

Expiration Date: Dec-2006
Page: 1 of 2

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
acetophenone	000098-86-2	BY02623AY	1003 ± 5 µg/mL
2-acetylaminofluorene	000053-96-3	04157CZ	1003 ± 5 µg/mL
4-aminobiphenyl	000092-67-1	LS15482LS	1003 ± 5 µg/mL
aniline	000062-53-3	3607BM	1003 ± 5 µg/mL
benzyl alcohol	000100-51-6	JN11713BN	1003 ± 5 µg/mL
2,6-dichlorophenol	000087-65-0	MS03518LN	1003 ± 5 µg/mL
p-(dimethylamino)azobenzene	000060-11-7	DN24537DN	1003 ± 5 µg/mL
7,12-dimethylbenz[a]anthracene	000057-97-6	09516HS	1003 ± 5 µg/mL
m-dinitrobenzene (1,3)	000099-65-0	180-148A	1003 ± 5 µg/mL
dinoseb	000088-85-7	29550-47	1003 ± 5 µg/mL
diphenylamine	000122-39-4	JY02413HY	1003 ± 5 µg/mL
ethyl methanesulfonate	000062-50-0	TR09224PR	1003 ± 5 µg/mL
hexachloropropene	001888-71-7	EF02811EF	1003 ± 5 µg/mL
isosafrole	000120-58-1	HY11325HX	1003 ± 5 µg/mL
3-methylcholanthrene	000056-49-5	15122EO	1003 ± 5 µg/mL
methyl methanesulfonate	000066-27-3	CF14822BF	1003 ± 5 µg/mL
m-cresol	000108-39-4	BG03517AG	1003 ± 5 µg/mL
1-naphthylamine	000134-32-7	05138AS	1003 ± 5 µg/mL
2-naphthylamine	000091-59-8	01635CU	1003 ± 5 µg/mL
N-nitrosodi-n-butylamine	000924-16-3	80H7706	1003 ± 5 µg/mL
N-nitrosodiethylamine	000055-18-5	FCL02	1003 ± 5 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

- NM in ETS database.

Quality Endorsed Company ISO 9001 SAI Global Registered

ACCREDITED ISO 17025 Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

Uncontrolled Document

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Semi-Volatiles Mixture

Product SVM-8271
Lot Number: CB-2681

Expiration Date: Dec-2006

Analyte	CAS#	Analyte Lot	True Value
N-nitrosomethylethylamine	010595-95-6	26H104	1003 ± 5 µg/mL
N-nitrosomorpholine	000059-89-2	FII01	1003 ± 5 µg/mL
N-nitrosopiperidine	000100-75-4	50H04081	1003 ± 5 µg/mL
N-nitrosopyrrolidine	000930-55-2	MX04025BM	1003 ± 5 µg/mL
5-nitro-o-toluidine	000099-55-8	AR03028KN	1003 ± 5 µg/mL
pentachlorobenzene	000608-93-5	JW0062627	1003 ± 5 µg/mL
pentachloroethane	000076-01-7	337-108A	1003 ± 5 µg/mL
pentachloronitrobenzene	000082-68-8	TQ08020EQ	1003 ± 5 µg/mL
phenacetin	000062-44-2	JN14230TG	1003 ± 5 µg/mL
safrole	000094-59-7	EY13103EY	1003 ± 5 µg/mL
1,2,4,5-tetrachlorobenzene	000095-94-3	N950075	1003 ± 5 µg/mL
2,3,4,6-tetrachlorophenol	000058-90-2	30319-21	1003 ± 5 µg/mL
o-toluidine	000095-53-4	200-17C	1003 ± 5 µg/mL
1,3,5-trinitrobenzene	000099-35-4	N990079	1002 ± 5 µg/mL

Matrix: methylene chloride (dichloromethane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

8270 55 Mix3 (3 of 5) CUS-7266 (8 Ampules)
6A31046
6A31049 → 55

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Custom Standard

Product CUS-7266 **Expiration Date:** Feb-2008
Lot Number: CC-0160 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
biphenyl	000092-52-4	NT01492	2004 ± 10 µg/mL
benzoic acid	000065-85-0	JM-00103JM	2004 ± 10 µg/mL
pyridine	000110-86-1	00115DM	2008 ± 10 µg/mL
benzaldehyde	000100-52-7	CZ-03625BZ	2008 ± 10 µg/mL
e-caprolactam	000105-60-2	DR-06716LQ	2004 ± 10 µg/mL

Matrix: methylene chloride (dichloromethane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
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ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

8270 SIM Mix 4 (Y of 5) CUS-7270

6A31057

6A31072-78

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

Custom Standard

Product	CUS-7270	**Expiration Date:**	Feb-2007
Lot Number:	CC-0195	**Page:**	1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
atrazine	001912-24-9	EB-1940	2004 ± 10 µg/mL

Matrix: acetone

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851-01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

8270 SS Mix 5 (5 cl) US-105N

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

6A31058 - 63
+
6A31064 — 69

Benzidines Mixture

Product US-105N
Lot Number: CB-0430

Expiration Date: Mar-2007
Page: 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
benzidine	000092-87-5	RAO18	2006 ± 10 µg/mL
3,3'-dichlorobenzidine	000091-94-1	FCW01	2006 ± 10 µg/mL

Matrix: methanol (methyl alcohol)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company
ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Edward Fitzgerald

Dr. Edward Fitzgerald,
Senior Scientist

GA09094 – GA09117

ULTRA SCIENTIFIC
Analytical Solutions

Certificate of Analysis

2-Fluorobiphenyl Solution

Product ATS-140 **Expiration Date:** Oct-2008

Lot Number: CA-1690 **Page:** 1 of 1

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

Analyte	CAS#	Analyte Lot	True Value
2-fluorobiphenyl	000321-60-8	06511CB	2001 ± 10 µg/mL

Matrix: methylene chloride (dichloromethane)

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.

Quality System
Quality Endorsed Company ISO 9001
SAI Global Registered

ACCREDITED
ISO 17025
Cert. No. 0851- 01

250 Smith Street, North Kingstown, RI 02852 USA
401-294-9400 Fax: 401-295-2330
www.ultrasci.com

Dr. Edward Fitzgerald,
Senior Scientist

GA09080 – GA09084

AccuStandard Inc.
125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: DRH-006S
DESCRIPTION: Proposed DEP(MA) - PAH Mix
LOT: B6010025
SOLVENT: CH2Cl2

EXPIRATION: Jan 4, 2016

See reverse for additional certification information.

This product is guaranteed accurate to + 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/MS)	Prepared Concentration[1] (µg/mL)		Certified Analyte Concentration[2] (µg/mL)
Acenaphthene	83-32-9	99.3	1001	±40.04	994
Acenaphthylene	208-96-8	98.5	1002	±40.08	987
Anthracene	120-12-7	100	1001	±40.04	1001
Benz(a)anthracene	56-55-3	99.5	1001	±40.04	996
Benzo(a)pyrene	50-32-8	98.3	1001	±40.04	984
Benzo(b)fluoranthene	205-99-2	99.9	1000	±40.00	999
Benzo(g,h,i)perylene	191-24-2	98	1001	±40.04	981
Benzo(k)fluoranthene	207-08-9	99.1	1001	±40.04	992
Chrysene	218-01-9	99.5	1000	±40.00	995
Dibenz(a,h)anthracene	53-70-3	99.5	1000	±40.00	995
Fluoranthene	206-44-0	99.9	1001	±40.04	1000
Fluorene	86-73-7	100	1001	±40.04	1001
Indeno(1,2,3-cd)pyrene	193-39-5	100	1000	±40.00	1000
2-Methylnaphthalene	91-57-6	98.8	1001	±40.04	989
Naphthalene	91-20-3	99.8	1001	±40.04	999
Phenanthrene	85-01-8	98.4	1001	±40.04	985
Pyrene	129-00-0	98.6	1001	±40.04	987

17 Components

1. All weights are traceable through NIST, Test No.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty u,(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is U,(y) *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-001
Rev. 11/02

Certificate of Analysis

6409078

DESCRIPTION: EPA 8270 GC/MS Tuning Solution

CATALOG NO.: 47387 MFG DATE: May-2005

LOT NO.: LB30314 EXPIRATION DATE: May-2008

SOLVENT: METHYLENE CHLORIDE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY(2)	WEIGHT(3) CONCENTRATION	ANALYTICAL(4) CONCENTRATION		STD DEV	SUPELCO LOT NO
BENZIDINE	92-87-5	99.4	50.10	51.27	+/-	0.347	LA42637
DFTPP	5074-71-5	97.5	50.12	51.66	+/-	0.643	LB24797
PENTACHLOROPHENOL	87-86-5	99.9	50.20	53.05	+/-	0.962	LB01443
4,4'-DDT	50-29-3	98.9	50.20	52.12	+/-	1.040	LA83013

(1) Listed in alphabetical order.

(2) Detemined by capillary GC-FID, unless otherwise noted.

(3) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.

(4) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.

Elwood Doughty
Quality Control Supervisor

SUPELCO
595 North Harrison Road
Bellefonte, PA 16823-0048 USA
Phone (814) 359-3441

LB15085-86

CERTIFICATE OF ANALYSIS

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800) 356-1688
Fax: (814) 353-1309

Catalog No.: 31285 **Lot No.:** A040537

Description: 2-Methylnaphthalene Standard

Expiration Date[1]: April 2012 **Storage:** Refrigerate

Elution Order	Compound	CAS#	Percent Purity[2]	Concentration[3]	Percent Uncertainty[4]
1	2-Methylnaphthalene	91-57-6	98%	1000 ug/mL	+/- 0.04
	Solvent: Methylene Chloride	75-09-2	99%		

Column:
30m x .25mm x .5um
Rtx-5 (cat.# 10238)

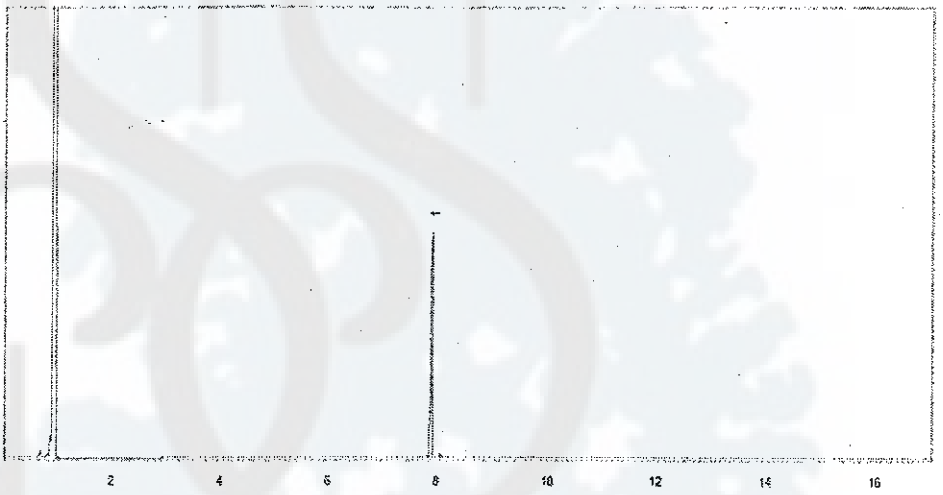
Carrier Gas:
hydrogen @ 40 cm/sec

Temp. Program:
40°C to 300°C @ 15°C/min.

Inj. Temp:
250°C

Det. Temp:
300°C

Det. Type:
FID



Manufactured By: RM

John Lidgett - QA Analyst

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addtion to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reftactive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NISTtraceable weights (7 mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured Under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

TOPLEVEL PARAMETERS

(GC/MS-1)

Method Information For: C:\HPCHEM\1\METHODS\SV1NB.M

Method Sections To Run:

Appendix C

( ) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Method Comments:
THIS METHOD IS USED TO ANALYZE FOR BASE-NEUTRAL AND ACID EXTRACTABLE COMPOUNDS USING A CAPILLARY COLUMN. THIS METHOD IS USING TCL SOW 88 LIST. 625 AND 8270 ARE APPLICABLE 02/95.

END OF TOPLEVEL PARAMETERS

ACQUISITION PARAMETERS

General Information
------- -----------

Inlet : GC
Tune File : DFTPP.U
Acquisition Mode : Scan

MS Information
-- -----------

Solvent Delay : 0.75 min

EM Absolute : False
EMV Offset : 0.0
Resulting Voltage : 1835.3

[Scan Parameters]

Low Mass : 35
High Mass : 500
Threshold : 500
Sampling # : 2 A/D Samples 4
Plot 1 low mass : 35
Plot 1 high mass: 500

[Real Time Plot Parameters]

Time Window : 25 min
Iconize Real Time Display : False
Plot 1 type : Extracted ion
Scale minimum : 0
Scale maximum : 400000
Plot 2 type : No plot

GC Inlet Information
-- ----- -----------

[Inlet A Temperature Program Information]

Oven Track : Off
Inlet A Off

[Inlet B Temperature Program Information]

Oven Track : Off
Initial Temp. : 275 C
Initial Time : 480.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Inlet A Pressure Program Information]

Constant Flow : On 7.1 psi at 40 C
Pressure Units : psi

[Inlet A Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 8.0 psi
Flow : 1.1 ml/min
Linear velocity : 37.5 cm/sec
Split flow : 1 ml/min
Split ratio : 0.9

[Inlet B Pressure Program Information]

Constant Flow : Off
Initial Pres. : 7.1 psi
Initial Time : 0.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	99.00	36.0	0.70
2	99.00	7.1	0.00
3	0		

Total Program Time: 1.28 min
Pressure Units : psi

[Inlet B Flow Settings]

Column length : 30.00 m
Column diameter : 0.250 mm
Gas : He
Vacuum compensation : On
Pressure : 7.1 psi
Flow : 0.9 ml/min
Linear velocity : 33.9 cm/sec
Split flow : 1 ml/min
Split ratio : 1.2

[Auxiliary Channel C Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)

```
  1               0
Total Program Time: 480.00 min
```

[Auxiliary Channel D Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel E Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

[Auxiliary Channel F Information]

Comment:

Pressure Program:
Initial Pres. : 0.0 psi
Initial Time : 480.00 min

Level	Rate(psi/min)	Final Pres.(psi)	Final Time (min)
1	0		

Total Program Time: 480.00 min

GC Temperature Information
-- ----------- -----------

[GC Zone Temperatures]

Inj. A : 250 C Off
Inj. B : 275 C
Det. A : 50 C Off
Det. B : 300 C
Aux.   : 280 C Off

[Oven Parameters]

Oven Equib Time : 0.20 min
Oven Max   : 325 C
Oven   : On
Cryo   : Off
Ambient   : 25 C
Cryo Blast   : Off

[Oven Program]

Initial Temp. : 60 C
Initial Time : 2.00 min

Level	Rate (C/min)	Final Temp. (C)	Final Time (min)
1	35.00	130	0.00
2	12.00	325	3.75
3	0.00		

Next Run Time : 24.00 min

Injector Information
--- ---

Injection Source : Auto
Injection Location : Front

Sample Washes : 1
Sample Pumps : 4
Sample Volume : 1 stop(s)
Viscosity Delay : 1 sec
Solvent A Washes : 4
Solvent B Washes : 2
On Column : No

[Purge Information]

Purge A/B	Init. Value	On Time	Off Time
A	On	0.15	0.00
B	On	0.15	0.00

END OF ACQUISITION PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\SV1NB.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: Yes
Printer: No
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
C:\DATABASE\NBS75K.L	0
C:\DATABASE\PAH.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Detailed

Output Destination
    Screen: No
    Printer: Yes
    File: No

Generate Report During Run Method: No

ELEMENT ID: 0603035(SOIL) 0603036(AQUEOUS)
Calibration Last Updated: Fri Apr 07 09:13:18 2006

Reference Window: 5.00 Percent
Non-Reference Window: 5.00 Percent
Correlation Window: 0.03 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD)

Ret. Time 3.86 min., Extract & Integrate from 3.36 to 4.36 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	152.00			*** METH DEFAULT ***
Q1	115.00	58.70	30.0	*** METH DEFAULT ***
Q2	150.00	289.20	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response
5		40.000	442167
10		40.000	372682
50		40.000	381573
80		40.000	339990
120		40.000	337070
160		40.000	306591
200		40.000	356509
25		40.000	437292
CC		40.000	499862

Qualifier Peak Analysis ON ISTD conc: 40.000 ng/uL
Curve Fit: Avg. RF

---

2) N-Nitrosodimethylamine ( )

Ret. Time 0.85 min., Extract & Integrate from 0.35 to 1.35 min.

Signal		Rel Resp.	Pct. Unc.(abs)	Integration
Tgt	74.05			*** METH DEFAULT ***
Q1	42.10	53.30	30.0	*** METH DEFAULT ***

Lvl	ID	Conc (ng/uL)	Response

TOPLEVEL PARAMETERS

Method Information For: C:\HPCHEM\1\METHODS\SV2KC.M

Method Sections To Run:

(X) Save Copy of Method With Data
( ) Pre-Run Cmd/Macro =
(X) Data Acquisition
( ) Data Analysis
( ) Post-Run Cmd/Macro =

Appendix C

Method Comments:
8270

END OF TOPLEVEL PARAMETERS

INSTRUMENT CONTROL PARAMETERS (MS2)

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

7673 Injector

Front Injector:
No parameters specified

Back Injector:

Sample Washes	2
Sample Pumps	2
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanoliter Adapter	Off
PostInj Solvent A Washes	2
PostInj Solvent B Washes	2
Viscosity Delay	1 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	275 C
Detector A:	Off	50 C
Detector B:	On	300 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equib Time:	0.10 minutes
Oven Max:	350 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature: 60 C
Initial Time: 1.00 minutes

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	35.0	130	0.00
2(A)	12.0	325	4.75
3(B)	0.0	50	1.00

Next Run Time: 24.00 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow: Off
Constant Flow Pressure: 0.0 psi
Constant Flow Temperature: 50 C
Initial Pressure: 0.0 psi
Initial Time: 650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length: 30.00 m
Column Diameter: 0.530 mm
Gas: He
Vacuum Compensation: Off

Inlet B:

Constant Flow: Off
Constant Flow Pressure: 0.0 psi
Constant Flow Temperature: 50 C
Initial Pressure: 7.0 psi
Initial Time: 0.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	99.00	36.0	0.50
2(A)	99.00	7.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 1.09 minutes

Column Length: 30.00 m
Column Diameter: 0.250 mm
Gas: He
Vacuum Compensation: On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge Init Value On Time Off Time Splitless Injection

A	Off	0.17	0.00	No
B	On	0.17	0.00	No

HP5890 Valve and Relay Information

Initial Setpoints:

5890 Valves:

Valve 1: Off Valve 2: Off Valve 3: Off Valve 4: On

19405 Valves:

Valve 5: Off Valve 6: Off Valve 7: Off Valve 8: Off

19405 Relays:

Relay 1: Off Relay 2: Off Relay 3: Off Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Comp1	0.053	5.000	0.00	1.00
2	Testplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETERS

General Information

Tune File : DFTPP.U
Acquistion Mode : Scan

MS Information

Solvent Delay : 1.20 min

EM Absolute : False
EM Offset : 0
Resulting EM Voltage : 2788.2

[Scan Parameters]

Low Mass : 35
High Mass : 500
Threshold : 500
Sample # : 2 A/D Samples 4
Plot 2 low mass : 50
Plot 2 high mass : 550

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

DATA ANALYSIS PARAMETERS

Method Name: C:\HPCHEM\1\METHODS\PAH2DR.M

Percent Report Settings

Sort By: Signal

Output Destination
Screen: No
Printer: Yes
File: No

Integration Events: Meth Default

Generate Report During Run Method: No

Signal Correlation Window: 0.020

Qualitative Report Settings

Peak Location of Unknown: Apex

Library to Search	Minimum Quality
nbs75k.L	0

Integration Events: Meth Default

Report Type: Summary

Output Destination
Screen: Yes
Printer: No
File: No

Generate Report During Run Method: No

Quantitative Report Settings

Report Type: Summary

Output Destination
Screen: Yes
Printer: Yes
File: No

Generate Report During Run Method: No

LL PAH ELEMENT ID 0604005
Calibration Last Updated: Thu Apr 06 15:54:57 2006

Reference Window: 0.50 Minutes
Non-Reference Window: 0.50 Minutes
Correlation Window: 0.02 minutes
Default Multiplier: 1.00
Default Sample Concentration: 0.00

Compound Information

---

1) 1,4-Dichlorobenzene-d4 (ISTD TR)

Ret. Time 4.21 min., Extract & Integrate from 4.06 to 4.36 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 152.00			*** METH DEFAULT ***
Q1 115.00	51.80	30.0	*** METH DEFAULT ***
Q2 150.00	153.70	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ng/uL)	Response
0.2	2.000	5361
0.4	2.000	5216
1.0	2.000	4812
2.0	2.000	10368
5.0	2.000	5671
8.0	2.000	5719
CC	2.000	6400
0.1	2.000	5740

Qualifier Peak Analysis ON ISTD conc: 2.000 ng/uL
Curve Fit: Avg. RF

---

2) 1,2 Dichlorobenzene-d4(SURR) ( )

Ret. Time 4.41 min., Extract & Integrate from 3.91 to 4.91 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 152.00			*** METH DEFAULT ***
Q1 150.00	173.80	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ng/uL)	Response
0.2	0.200	588
0.4	0.400	1046
1.0	1.000	2405
2.0	2.000	10416
5.0	5.000	13918
8.0	8.000	23368
CC	1.000	-1
0.1	0.100	384

Qualifier Peak Analysis ON
Curve Fit: Avg. RF

---

3) Naphthalene-d8 (ISTD TR)

Ret. Time 5.67 min., Extract & Integrate from 5.52 to 5.82 min.

Signal	Rel Resp.	Pct. Unc.(rel)	Integration
Tgt 136.00			*** METH DEFAULT ***
Q1 68.00	7.10	30.0	*** METH DEFAULT ***

Lvl ID	Conc (ng/uL)	Response
0.2	2.000	13794
0.4	2.000	14266
1.0	2.000	13382

Appendix D

ESS Laboratory (SVOA)
MS1 Sequence Log

Work Orders Reported: _____

Date Analyzed: _____ Analyst: _____

	8270/625	Seq ID	Calibration ID
Soil / Aqueous			

	Other Method:	Seq ID	Calibration ID
Soil / Aqueous			

** Note in comments when other method used.*

Vial	File	Lab Id	Rw	Rpt	Imp By	STD ID/ Comments	Batch ID	Matrix	-RE Ref
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
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	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								
	SV1								

ICAL/CCAL Failures, Quads: _____

Comments: _____

Tune Method: DFTPP

ICAL Batch ID: BB71420(Aq); BB71421(Soil)

Final Data Review: _____

Signed copy included with each work order.

ISTD ID : _____

Lobgook Review: _____

Control #60.0044-0701A

Appendix D.

ESS Laboratory (SVOA)
MS2 Sequence Log

Work Orders Reported: _____

Date Analyzed: _____ Analyst: _____

	8270/625	Seq ID	Calibration ID
Soil / Aqueous			

	Other Method:	Seq ID	Calibration ID
Soil / Aqueous			

** Note in comments when other method used.*

Vial	File	Lab Id	Rw	Rpt	Imp By	STD ID/ Comments	Batch ID	Matrix	-RE Ref
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
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	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								
	SV2								

ICAL/CCAL Failures, Quads: _____

Comments: _____

Tune Method: DFTPP

ICAL Batch ID: BB71420(Aq); BB71421(Soil)

Final Data Review: _____

Signed copy included with each work order.

ISTD ID : _____

Control #60.0043-0702A

Lobgook Review: _____

Attachment D
SOP 60_8270

ESS LABORATORY
GC/MS 1 MAINTENANCE RECORD

Date	Trouble Observed	Service Done	Initials

All maintenance must be recorded.
Column serial #s and new parts must be noted.

Control #60.0021-0501A

Page_____

Reviewed by _____

Attachment E

Lab Data Review Check List
METHOD 8270C (GC/MS)

Project Number(s):			
Batch Number (s):	Method:		
Review Item	**Yes (x)**	**No (x)**	**N/A (x)**
1. Tuning a) Were all 8270 samples run within 12 hours of a DFTPP tuning and were criteria met?			
2. Initial Calibration a) Does calibration curve consist of minimum of 5 point for average RF or LR, minimum of 6 points for QR?			
b) Is the low standard at or below the MRL?			
c) Are the RRFs and the % RSDs within QC limits for appropriate analytes?			
3. Second Source Validation a) Was the initial calibration curve verified by a second source calibration standard (ICV)?			
4. Continuing Calibration a) Is the Continuing Calibration Verification (CCV) standard run every 12 hours?			
b) Are the RRFs and % differences within QC limits for the CCC and SPCCs?			
5. Sample Analysis a) Are sample holding times being met? Soils- 14 day, Aqueous- 7 days			
b) Was pH checked and recorded for all samples?			
c) Are all samples with concentrations > the highest standard used for initial calibration, diluted and re-analyzed?			
d) RRT of identified compounds within ± 0.06 RRT units of RRT of standard component?			
e) Ions present in the standard spectra within abundance of >10% of the base ion present in sample spectra? Do abundance agree to 30% as compared to standard spectra? Was the mass-spectra visually reviewed and compared to the reference spectrum for all hits?			
f) Are Internal Standard areas within 50-200% of CCV?			
g) Are surrogate recoveries within QC limits?			
h) Are samples quantified against the appropriate internal standard?			
7. QC Samples a) Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the ½ MRL?			
b) Are the Laboratory Control Sample and its percent recovery within QC limits?			
c) Is the Matrix Spike/Matrix Spike Duplicate run at the desired frequency and is the percent recovery/RPD within QC limits?			
8. Others a) Are all non-conformances included and noted?			
b) Are all calculations checked at the minimum frequency?			
c) Did analyst sign/date the appropriate printouts and report sheets?			
d) Are sample IDs and units checked for transcription errors?			

Comments on any "No" responses:_____

_____

_____

Samples	Dilution	Instr.	Sequence	Directory

Analyst: _____ Date:_____ 2nd Level Rvw :_____ Date: _____

Appendix F

ESS Laboratory Ccal/Batch Exception sheet
Semi-VOLATILE ORGANICS LABORATORY (GC/MS)

Project Number(s): _____ Instrument ID: _____ **CCal ID/date:** _____

Target Analytes	CCV	BS/ BSD	MS/ MSD	Target Analytes	CCV	BS/ BSD	MS/ MSD	Target Analytes	CCV	BS/ BSD	MS/ MSD
N-Nitrosodimethylamine				4-Chloroaniline				N-nitrosodiphenylamine			
Pyridine				Hexachlorobutadiene				Azobenzene			
bis(2-Chloroethyl)ether				4-Chloro-3-Methylphenol				4-Bromophenyl-phenylether			
2-Chlorophenol				2-Methylnaphthalene				Hexachlorobenzene			
Phenol				1-Methylnaphthalene				Pentachlorophenol			
Aniline				Hexachlorocyclopentadiene				Phenanthrene			
1,3-Dichlorobenzene				2,4,6-Trichlorophenol				Anthracene			
1,4-Dichlorobenzene				2,4,5-Trichlorophenol				Carbazole			
1,2-Dichlorobenzene				Biphenyl				Di-n-butylphthalate			
Benzyl Alcohol				2-Chloronaphthalene				Fluoranthene			
bis(2-chloroisopropyl)Ether				Dimethylphthalate				Benzidine			
2-Methylphenol				Acenaphthylene				Pyrene			
Acetophenone				2,6-Dinitrotoluene				Butylbenzylphthalate			
N-Nitroso-Di-n-Propylamine				2-Nitroaniline				3,3'-Dichlorobenzidine			
Hexachloroethane				Acenaphthene				Benzo(a)anthracene			
3+4-Methylphenol				2,4-Dinitrophenol				Chrysene			
Nitrobenzene				Dibenzofuran				bis(2-Ethylhexyl)phthalate			
Isophorone				4-Nitrophenol				Di-n-octylphthalate			
2-Nitrophenol				3-Nitroaniline				Benzo(b)fluoranthene			
Benzoic Acid				2,4-Dinitrotoluene				Benzo(k)fluoranthene			
2,4-Dimethylphenol				Fluorene				Benzo(a)pyrene			
bis(2-Chloroethoxy)methane				Diethylphthalate				Indeno(1,2,3-Cd)Pyrene			
2,4-Dichlorophenol				4-Chloro-phenyl-phenyl ether				Dibenzo(a,h)Anthracene			
1,2,4-Trichlorobenzene				4-Nitroaniline				Benzo(g,h,i)perylene			
Naphthalene				4,6-Dinitro-2-Methylphenol							

Initials and date: _____

Mark compounds that exceed criteria either Low (L) or High (H) bias

CCCs must be < 20%, all other analytes < 30% drift or difference.

SPCCs must be ≥ method minimum (0.05).

Batch ID(s)

Batch#1: _____

Batch#2: _____

Batch#3: _____

Tune	P/F
DDT breakdown	
Tailing	

P = pass, F = Fail